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=> file reg
FILE 'REGISTRY' ENTERED AT 12:07:40 ON 12 JUN 2003
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=> display history full l1-


     FILE 'REGISTRY' ENTERED AT 11:01:44 ON 12 JUN 2003
                    E AMMONIA/CN
L1            1 SEA AMMONIA/CN
                    E POTASSIUM NITRATE/CN
L2            1 SEA "POTASSIUM NITRATE"/CN
                    E SODIUM NITRATE/CN
L3            1 SEA "SODIUM NITRATE"/CN
                    E SODIUM HYDROXIDE/CN
L4            1 SEA "SODIUM HYDROXIDE"/CN
                    E POTASSIUM HYDROXIDE/CN
L5            1 SEA "POTASSIUM HYDROXIDE"/CN

     FILE 'LCA' ENTERED AT 11:04:25 ON 12 JUN 2003
L6        32136 SEA (PRODUC? OR PROD# OR GENERAT? OR MANUF? OR MFR# OR
                CREAT? OR FORM## OR FORMING# OR FORMAT? OR MAKE# OR
                MADE# OR MAKING# OR FABRICAT? OR SYNTHESI? OR PREPAR? OR
                PREP#)/BI,AB

     FILE 'HCA' ENTERED AT 11:07:18 ON 12 JUN 2003
L7        48759 SEA L1/P OR (PRODUC? OR PROD# OR GENERAT? OR MANUF? OR
                MFR# OR CREAT? OR FORM## OR FORMING# OR FORMAT? OR MAKE#
                OR MADE# OR MAKING# OR FABRICAT? OR SYNTHESI? OR PREPAR?
                OR PREP#)(2A)(L1 OR AMMONIA# OR NH3)
L8        32713 SEA L2 OR (POTASSIUM# OR K)(W)NITRATE# OR KNO3
L9        34558 SEA L3 OR (SODIUM# OR NA)(W)NITRATE# OR NANO3
L10      213469 SEA L4 OR (POTASSIUM# OR K)(W)HYDROXIDE# OR KOH OR
                POTASH#
L11      393383 SEA L5 OR (SODIUM# OR NA)(W)HYDROXIDE# OR NAOH OR LYE#
                OR CAUSTIC# OR ENCAUSTIC#

     FILE 'LCA' ENTERED AT 11:11:32 ON 12 JUN 2003
L12        3729 SEA (ADDITIVE? OR RETARDER? OR IMPROVER? OR STABILIZER?
                OR STABILISER? OR INHIBITOR? OR MODIFIER? OR ACTIVATOR?
                OR DEACTIVATOR? OR APPRECIATOR? OR BOOSTER? OR SUPPRESSOR
                ? OR SCAVENGER? OR ENHANCER? OR ACCELERANT? OR ACCELERAT!
                R?)/BI,AB

     FILE 'HCA' ENTERED AT 11:13:38 ON 12 JUN 2003
L13        7649 SEA (COOL? OR CHILL?)(2A)(ADDITIVE? OR IMPROVER? OR
                STABILIZER? OR STABILISER? OR MODIFIER? OR ACTIVATOR? OR
                APPRECIATOR? OR BOOSTER? OR ENHANCER? OR ACCELERANT? OR
                ACCELERAT!R? OR AGENT? OR SALT#)/BI,AB
L14          50 SEA L7 AND L13
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L15              1 SEA L14 AND L8
L16              2 SEA L14 AND L9
L17              4 SEA L14 AND L10
L18              4 SEA L14 AND L11
L19              2 SEA L14 AND L10 AND L11
L20              7 SEA (L15 OR L16 OR L17 OR L18 OR L19)
L21            268 SEA L7 AND L8
L22            249 SEA L7 AND L9
L23            914 SEA L7 AND L10 AND L11
L24         629037 SEA COOL? OR CHILL?
L25             19 SEA L21 AND L24
L26             17 SEA L22 AND L24
L27            181 SEA L23 AND L24
L28                QUE CAT# OR CATALY?
L29             30 SEA L27 AND L28
L30          10568 SEA (COOL? OR CHILL?)(2A)(EFFLUEN? OR STREAM? OR FLOW OR
                   FLOWS OR FLOWED OR FLOWING#)
L31              2 SEA L25 AND L30
L32              1 SEA L26 AND L30
L33              2 SEA L27 AND L30
L34              1 SEA L29 AND L30
L35              3 SEA (L31 OR L32 OR L33 OR L34)
L36           3019 SEA CONDENS?(3A)(L1 OR AMMONIA# OR NH3)
L37              0 SEA L25 AND L36
L38              0 SEA L26 AND L36
L39              3 SEA L27 AND L36
L40              1 SEA L29 AND L36
L41              3 SEA L39 OR L40
L42            268 SEA L7 AND L8
L43            249 SEA L7 AND L9
L44            914 SEA L7 AND L10 AND L11
L45              3 SEA (L42 OR L43 OR L44) AND L30
L46             16 SEA (L42 OR L43 OR L44) AND L36
L47              6 SEA L46 AND L28
L48              3 SEA (L42 OR L43 OR L44) AND L13
L49            209 SEA (L42 OR L43 OR L44) AND L24
L50            311 SEA (L42 OR L43 OR L44) AND L28
L51             39 SEA L49 AND L50

     FILE 'LCA' ENTERED AT 11:28:49 ON 12 JUN 2003
L52           2452 SEA (RECOVER? OR RECLAMAT? OR RECLAIM? OR RETRIEV? OR
                   SALVAG? OR REGENERAT? OR RECONDITION? OR REFORM? OR
                   RECONSTITUT? OR REUSE# OR REUSING# OR RECYCL? OR
                   REPROCESS?)/BI,AB
L53              4 SEA (RE(W)(COVER? OR CLAMAT? OR CLAIM? OR GENERAT? OR
                   CONDITION? OR FORM? OR CONSTITUT? OR USE# OR USING# OR
                   CYCL? OR PROCESS?))/BI,AB

     FILE 'HCA' ENTERED AT 11:30:17 ON 12 JUN 2003
L54           2380 SEA (L52 OR L53)(5A)(SYN OR SYNTH?)(3A)(GAS## OR
                   GASEOUS? OR GASIF?)
L55              3 SEA (L42 OR L43 OR L44) AND L54
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L56            18 SEA L20 OR L35 OR L41 OR L45 OR L47 OR L48 OR L55
L57            40 SEA (L25 OR L26 OR L46) NOT L56
L58            27 SEA (L29 OR L51) NOT (L56 OR L57)
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=> file hca
FILE 'HCA' ENTERED AT 12:07:55 ON 12 JUN 2003
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=> d l56 1-18 cbib abs hitstr hitind

L56 ANSWER 1 OF 18 HCA COPYRIGHT 2003 ACS
134:147499 Method for preparation of 2,2,6,6-tetramethyl-4-oxopiperidine. Kadota, Yoichi; Fujii, Takeo (Sumitomo Chemical Co., Ltd., Japan). Jpn. Kokai Tokkyo Koho JP 2001031651 A2 20010206, 5 pp. (Japanese). CODEN: JKXXAF. APPLICATION: JP 1999-201633 19990715.

AB A crude reaction soln. obtained by reacting at least one of acetone or its **condensate** with **NH3** in the presence of a **catalyst** or one obtained by reaction of 2,2,4,4,6-pentamethyl-2,3,4,5-tetrahydropyrimidine in the presence of water is treated with an aq. strong alkali soln. to ext. the **catalyst** from the oil layer, followed by washing the oil layer with an aq. weak alkali soln. and distn. of the oil layer to give 2,2,6,6-tetramethyl-4-oxopiperidine (I). The weak alkali is selected from Na2CO3, NaHCO3, KHCO3, and K2CO3. This process suppresses thermal decompn. of I during distn. and gives I of high purity in high yield.

IT **1310-73-2**, **Sodium hydroxide**, reactions
7664-41-7, **Ammonia**, reactions
(**prepn**. of tetramethyloxopiperidine from crude reaction soln. by **catalyst** extn. with aq. strong alkali soln., washing with aq. weak alkali soln., and distn.)

RN 1310-73-2 HCA
CN Sodium hydroxide (Na(OH)) (9CI) (CA INDEX NAME)

Na—OH

RN 7664-41-7 HCA
CN Ammonia (8CI, 9CI) (CA INDEX NAME)

NH_3

IC ICM C07D211-74
CC 27-16 (Heterocyclic Compounds (One Hetero Atom))
IT Distillation

Extraction
(prepn. of tetramethyloxopiperidine from crude reaction soln. by **catalyst** extn. with aq. strong alkali soln., washing with aq. weak alkali soln., and distn.)

IT 12125-02-9, Ammonium chloride, uses
(prepn. of tetramethyloxopiperidine from crude reaction soln. by **catalyst** extn. with aq. strong alkali soln., washing with aq. weak alkali soln., and distn.)

IT 826-36-8P, 2,2,6,6-Tetramethyl-4-oxopiperidine
(prepn. of tetramethyloxopiperidine from crude reaction soln. by **catalyst** extn. with aq. strong alkali soln., washing with aq. weak alkali soln., and distn.)

IT 144-55-8, Sodium bicarbonate, uses 298-14-6, Potassium bicarbonate 497-19-8, Sodium carbonate, uses 584-08-7, Potassium carbonate
(prepn. of tetramethyloxopiperidine from crude reaction soln. by **catalyst** extn. with aq. strong alkali soln., washing with aq. weak alkali soln., and distn.)

IT 67-64-1, Acetone, reactions 556-72-9, 2,2,4,4,6-Pentamethyl-2,3,4,5-tetrahydropyrimidine **1310-73-2**, **Sodium hydroxide**, reactions **7664-41-7**, **Ammonia**, reactions 19496-14-1, Acetone dimer
(**prepn**. of tetramethyloxopiperidine from crude reaction soln. by **catalyst** extn. with aq. strong alkali soln., washing with aq. weak alkali soln., and distn.)

L56 ANSWER 2 OF 18 HCA COPYRIGHT 2003 ACS

132:323640 Process and converter for **ammonia production**. Speth, Christian (Haldor Topsoe A/S, Den.). PCT Int. Appl. WO 2000026139 A1 20000511, 15 pp. DESIGNATED STATES: W: AE, AL, AM, AT, AU, AZ, BA, BB, BG, BR, BY, CA, CH, CN, CR, CU, CZ, DE, DK, DM, EE, ES, FI, GB, GD, GE, GH, GM, HR, HU, ID, IL, IN, IS, JP, KE, KG, KP, KR, KZ, LC, LK, LR, LS, LT, LU, LV, MA, MD, MG, MK, MN, MW, MX, NO, NZ, PL, PT, RO, RU, SD, SE, SG, SI, SK, SL, TJ, TM, TR, TT, TZ, UA, UG, US, UZ, VN, YU, ZA, ZW, AM, AZ, BY, KG, KZ, MD, RU, TJ, TM; RW: AT, BE, BF, BJ, CF, CG, CH, CI, CM, CY, DE, DK, ES, FI, FR, GA, GB, GR, IE, IT, LU, MC, ML, MR, NE, NL, PT, SE, SN, TD, TG. (English). CODEN: PIXXD2. APPLICATION: WO 1999-EP8055 19991025. PRIORITY: DK 1998-1398 19981030.

AB **NH3** is **produced** by contacting an **NH3 synthesis** gas with an **NH3 synthesis catalyst** in a reaction zone in .gtoreq.1 **catalyst**-filled tubes, under continuous **cooling** of the reaction zone by heat conduction using a **coolant**, and withdrawing an NH3-rich effluent stream from the reaction zone. The converter comprises .gtoreq.1 **catalyst**-filled tubes for passing an **NH3 synthesis** gas and holding an **NH3 synthesis catalyst** in .gtoreq.1 reaction zone, where a **coolant stream** is passed through the shell side of the double concentric tube, around the **catalyst** tubes. An NH3 effluent can be withdrawn at an intermediate point between reaction zones with **recycling** of unconverted **synthesis gas**. The

coolants can be eutectic mixts. (melts) contg. **KNO3**
, **NaNO3**, and NaNO2, or **NaOH** and **KOH**.

IT **7664-41-7P, Ammonia, preparation**
(process and converter for **ammonia prodn.**)
RN 7664-41-7 HCA
CN Ammonia (8CI, 9CI) (CA INDEX NAME)

NH_3

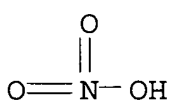
IT **1310-58-3, Potassium hydroxide**, uses
1310-73-2, Sodium hydroxide, uses
7631-99-4, Sodium nitrate, uses
7757-79-1, Potassium nitrate, uses
(process and converter for **ammonia prodn.**)
RN 1310-58-3 HCA
CN Potassium hydroxide (K(OH)) (9CI) (CA INDEX NAME)

K— OH

RN 1310-73-2 HCA
CN Sodium hydroxide (Na(OH)) (9CI) (CA INDEX NAME)

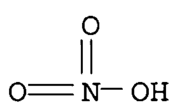
Na— OH

RN 7631-99-4 HCA
CN Nitric acid sodium salt (8CI, 9CI) (CA INDEX NAME)



Na

RN 7757-79-1 HCA
CN Nitric acid potassium salt (8CI, 9CI) (CA INDEX NAME)



K

IC ICM C01C001-04
CC 49-8 (Industrial Inorganic Chemicals)
Section cross-reference(s): 47, 67
ST **ammonia prodn** converter; reactor tubular **ammonia prodn**; converter **catalytic** **ammonia prodn**
IT Reactors
(**catalytic**; process and converter for **ammonia** **prodn.**)
IT **Salts**, uses
(molten, **coolants**; process and converter for **ammonia prodn.**)
IT Reactors
(tubular; process and converter for **ammonia** **prodn.**)
IT 7439-89-6, Iron, uses 7440-18-8, Ruthenium, uses
(process and converter for **ammonia prodn.**)
IT **7664-41-7P**, **Ammonia**, **preparation**
(process and converter for **ammonia prodn.**)
IT **1310-58-3**, **Potassium hydroxide**, uses
1310-73-2, **Sodium hydroxide**, uses
7631-99-4, **Sodium nitrate**, uses
7632-00-0, Sodium nitrite **7757-79-1**, **Potassium nitrate**, uses
(process and converter for **ammonia prodn.**)
IT 1333-74-0, Hydrogen, reactions 7727-37-9, Nitrogen, reactions
(process and converter for **ammonia prodn.**)

L56 ANSWER 3 OF 18 HCA COPYRIGHT 2003 ACS
132:127153 Recycling method of organic wastes containing PVC resins. Maeda, Sadahiko; Arao, Seiji; Suzuki, Katsutoshi (Ube Industries, Ltd., Japan). Jpn. Kokai Tokkyo Koho JP 2000044961 A2 20000215, 4 pp. (Japanese). CODEN: JKXXAF. APPLICATION: JP 1998-215004 19980730.
AB Org. wastes contg. PVC resins are treated by partial oxidn. in a gasification reactor to produce a raw synthesis gas mainly contg. H2, CO, O2 and water, wet scrubbing the raw synthesis gases with alkali aq. soln. contg. **NaOH** to trap the HCl formed during decompg. org. Cl compd. from the reactor, blowing and passing wastewaters soln. through a reverse-osmosis membrane module to sep. NaCl, discharging the concd. NaCl aq. soln. into a collecting tank, and then recycling the desalinated water into the scrubbing tower. The synthesis gas can be utilized as feedstocks in the **prodn** . of **NH3**, MeOH or O3. The method prevents dioxin formation and corrosion in process pipings.
IT **1310-73-2**, **Sodium hydroxide**, processes
(neutralization with; in reclamation of org. wastes contg. PVC resins by gasification)
RN 1310-73-2 HCA
CN Sodium hydroxide (Na(OH)) (9CI) (CA INDEX NAME)

Na—OH

IT **7664-41-7P**, **Ammonia**, processes
(**prodn**. feedstocks; from reclamation of org. wastes
contg. PVC resins by gasification)
RN 7664-41-7 HCA
CN Ammonia (8CI, 9CI) (CA INDEX NAME)

NH_3

IC ICM C10B053-00
ICS B09B003-00
CC 60-5 (Waste Treatment and Disposal)
Section cross-reference(s): 45, 49, 51
ST recycling org waste gasification PVC resin; **synthesis** gas
ammonia methanol **prodn** feedstock
IT **Synthesis gas** manufacturing
(for **recycling** method of org. wastes contg. PVC resins
by gasification)
IT **1310-73-2**, **Sodium hydroxide**, processes
(neutralization with; in reclamation of org. wastes contg. PVC
resins by gasification)
IT 67-56-1P, Methanol, processes **7664-41-7P**, **Ammonia**
, processes 10028-15-6P, Ozone, processes
(**prodn**. feedstocks; from reclamation of org. wastes
contg. PVC resins by gasification)
IT 630-08-0P, Carbon monoxide, processes 1333-74-0P, Hydrogen,
processes 7782-44-7P, Oxygen, processes
(**synthesis gas** contg.; from
reclamation of org. wastes contg. PVC resins by
gasification)

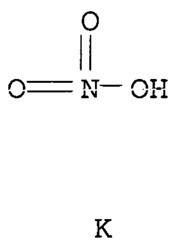
L56 ANSWER 4 OF 18 HCA
131:259559 Method for **ammonia production**. Whitlock,
David R. (USA). U.S. US 5968232 A 19991019, 12 pp., Cont.-in-part
of U. S. 5,676,737. (English). CODEN: USXXAM. APPLICATION: US
1997-898299 19970722. PRIORITY: US 1993-27890 19930308; US
1995-481800 19950607.
AB A process for the sepn. of ammonia dissolved in a gaseous solvent is
used to recover **ammonia** during the **prodn**. of
ammonia from a **synthesis** gas. The process may be
used to ext. and recover ammonia in either the supercrit. region or
the subcrit. region. The process can be used to remove
ammonia from **synthesis** gas, or other gaseous
solvents, by removing ammonia whose chem. potential decreases as the
d. of the solvent increases. The process utilizes a sorbent to
remove the ammonia followed by an in situ regeneration of the
sorbent and recovery of the ammonia. Typically, a gaseous solvent
contg. the ammonia is passed through a bed of sorbent to sorb the
ammonia onto the bed of sorbent, thereby producing a purified

gaseous solvent. At least a portion of the gaseous solvent is then acted upon to increase its solvent capacity for the ammonia. The increased solvent capacity gaseous solvent is passed through the bed of sorbent in the opposite direction to desorb the ammonia from the bed of sorbent to provide an increased solvent capacity gaseous solvent. The ammonia may then be recovered from the increased solvent capacity gaseous solvent.

IT **7757-79-1P, Potassium nitrate,**
preparation
(method for removing **ammonia** from **synthesis** gas or other gaseous solvents for manuf. of nitrogen-contg. fertilizers)

RN 7757-79-1 HCA

CN Nitric acid potassium salt (8CI, 9CI) (CA INDEX NAME)



IT **7664-41-7P, Ammonia, preparation**
(method for removing **ammonia** from **synthesis** gas or other gaseous solvents for manuf. of nitrogen-contg. fertilizers)

RN 7664-41-7 HCA

CN Ammonia (8CI, 9CI) (CA INDEX NAME)

NH_3

IC ICM B01D053-04

NCL 095090000

CC 49-8 (Industrial Inorganic Chemicals)
Section cross-reference(s): 19

ST **gaseous** solvent **synthesis gas ammonia recovery** fertilizer **manuf**

IT Solvents
Solvents
(gaseous; method for removing **ammonia** from **synthesis** gas or other gaseous solvents for manuf. of nitrogen-contg. fertilizers)

IT Gases
Sorbents
Synthesis gas
(method for removing **ammonia** from **synthesis** gas or other gaseous solvents for manuf. of nitrogen-contg. fertilizers)

IT Fertilizers
(method for removing **ammonia** from **synthesis** gas or other gaseous solvents for manuf. of nitrogen-contg. fertilizers)

IT Noble gases, processes
(method for removing **ammonia** from **synthesis** gas or other gaseous solvents for manuf. of nitrogen-contg. fertilizers)

IT Gases
Gases
(solvent; method for removing **ammonia** from **synthesis** gas or other gaseous solvents for manuf. of nitrogen-contg. fertilizers)

IT Aerogels
Molecular sieves
(sorbent; method for removing **ammonia** from **synthesis** gas or other gaseous solvents for manuf. of nitrogen-contg. fertilizers)

IT Salts, uses
Silica gel, uses
Zeolites (synthetic), uses
(sorbent; method for removing **ammonia** from **synthesis** gas or other gaseous solvents for manuf. of nitrogen-contg. fertilizers)

IT Chromatography
(supports, sorbent; method for removing **ammonia** from **synthesis** gas or other gaseous solvents for manuf. of nitrogen-contg. fertilizers)

IT 1344-28-1, Aluminum oxide (Al2O3), processes 7440-44-0, Carbon, processes
(activated, sorbent; method for removing **ammonia** from **synthesis** gas or other gaseous solvents for manuf. of nitrogen-contg. fertilizers)

IT 57-13-6P, Urea, preparation 6484-52-2P, Ammonium nitrate, preparation **7757-79-1P**, **Potassium nitrate**, preparation 7783-20-2P, Ammonium sulfate, preparation 10124-31-9P, Ammonium phosphate 10124-37-5P, Calcium nitrate
(method for removing **ammonia** from **synthesis** gas or other gaseous solvents for manuf. of nitrogen-contg. fertilizers)

IT **7664-41-7P, Ammonia, preparation**
(method for removing **ammonia** from **synthesis** gas or other gaseous solvents for manuf. of nitrogen-contg. fertilizers)

IT 74-82-8, Methane, processes 1333-74-0, Hydrogen, processes 7439-90-9, Krypton, processes 7440-01-9, Neon, processes 7440-37-1, Argon, processes 7440-59-7, Helium, processes 7440-63-3, Xenon, processes 7727-37-9, Nitrogen, processes
(method for removing **ammonia** from **synthesis** gas or other gaseous solvents for manuf. of nitrogen-contg. fertilizers)

L56 ANSWER 5 OF 18 HCA
127:35962 Nigrosine dyes and manufacture thereof. Maekawa, Yoshihiro (Maekawa, Yoshihiro, Japan). Jpn. Kokai Tokkyo Koho JP 09087535 A2 19970331 Heisei, 4 pp. (Japanese). CODEN: JKXXAF. APPLICATION: JP 1995-282342 19950925.

AB Dyes are prepd. by condensing aniline and nitrobenzene in the presence of HCl, FeCl2, etc., treating the liq. contg. the reaction **products** with **NH3**, sepg. the ppts. and the water phase, and distg. the oil layer to remove aniline and volatiles. Waste water contg. aniline, NH3, and/or ammonium salts is treated with **NaOH** and distd. to remove aniline and NH3.

IT **1310-73-2**, **Sodium hydroxide**, uses
7664-41-7, **Ammonia**, uses
(**condensation** of aniline with nitrobenzene in presence of **catalysts** for manuf. of nigrosine dyes and purifn.)

RN 1310-73-2 HCA
CN Sodium hydroxide (Na(OH)) (9CI) (CA INDEX NAME)

Na—OH

RN 7664-41-7 HCA
CN Ammonia (8CI, 9CI) (CA INDEX NAME)

NH_3

IC ICM C09B017-00
CC 41-5 (Dyes, Organic Pigments, Fluorescent Brighteners, and Photographic Sensitizers)
Section cross-reference(s): 61

IT Condensation reaction **catalysts**
(condensation of aniline with nitrobenzene in presence of **catalysts** for manuf. of nigrosine dyes and purifn.)

IT Dyes
(nigrosine; condensation of aniline with nitrobenzene in presence of **catalysts** for manuf. of nigrosine dyes and purifn.)

IT 7647-01-0, Hydrogen chloride, uses 7758-94-3, Iron chloride (FeCl2)
(condensation of aniline with nitrobenzene in presence of **catalysts** for manuf. of nigrosine dyes and purifn.)

IT 8005-03-6P, Nigrosine
(condensation of aniline with nitrobenzene in presence of **catalysts** for manuf. of nigrosine dyes and purifn.)

IT **1310-73-2**, **Sodium hydroxide**, uses
7664-41-7, **Ammonia**, uses
(**condensation** of aniline with nitrobenzene in presence of **catalysts** for manuf. of nigrosine dyes and purifn.)

IT 62-53-3, Aniline, reactions 98-95-3, Nitrobenzene, reactions
(condensation of aniline with nitrobenzene in presence of **catalysts** for manuf. of nigrosine dyes and purifn.)

L56 ANSWER 6 OF 18 HCA COPYRIGHT 2003 ACS
116:217444 Process and apparatus for the manufacture of water-insoluble, chainlike ammonium polyphosphate. Staffel, Thomas; Gradl, Reinhard; Becker, Wolfgang; Fucker, Gregor (Hoechst A.-G., Germany). Eur. Pat. Appl. EP 480180 A1 19920415, 7 pp. DESIGNATED STATES: R: AT, BE, CH, DE, DK, ES, FR, GB, IT, LI, NL, SE. (German). CODEN: EPXXDW. APPLICATION: EP 1991-115147 19910907. PRIORITY: DE 1990-4032133 19901010.

AB In this process, comprising reacting equimol. amts. of (NH4)2HPO4 and P2O5 at 170-350.degree. in a mixing-kneading-grinding zone in the presence of flowing NH3(g), the H2O(g) of the the H2O(g)-contg. NH3(g) emanating from that reaction zone is **condensed**, and the dry **NH3**(g) is recycled to the reaction zone. The vapors from the reaction zone enter a loop consisting of a **cooler**, pump, and heat exchanger, connected to NH3(g) feed. This arrangement avoids upsets of the biol. wastewater treatment facility, and the NH4 polyphosphate produced has pH 6.5, acid no. 0.3 mg **KOH**/g, and contains 4.3 wt.% water-sols. (at 25.degree.).

IT **1310-58-3P**, **Potassium hydroxide**, miscellaneous **1310-73-2P**, **Sodium hydroxide**, miscellaneous
(water vapor removal with, from ammonia-water vapor offgas from ammonium polyphosphate **manuf**., for **ammonia** recycle and decreased load on wastewater treatment facility, app. for)

RN 1310-58-3 HCA
CN Potassium hydroxide (K(OH)) (9CI) (CA INDEX NAME)

K—OH

RN 1310-73-2 HCA
CN Sodium hydroxide (Na(OH)) (9CI) (CA INDEX NAME)

Na—OH

IC ICM C01B025-40
CC 49-4 (Industrial Inorganic Chemicals)
ST ammonia recycle ammonium polyphosphate; diammonium phosphate phosphorus pentoxide ammonium polyphosphate; water vapor ammonia ammonium polyphosphate; **condenser** water vapor sepn **ammonia**; alkali metal hydroxide water vapor sepn

IT Condensation, physical
(water vapor removal by, from ammonia-water vapor offgas from ammonium polyphosphate **manuf**., for **ammonia** recycle and decreased load on wastewater treatment facility, app. for)

IT Alkali metal hydroxides
(water vapor removal with, from ammonia-water vapor offgas from

ammonium polyphosphate **manuf**., for **ammonia** recycle and decreased load on wastewater treatment facility, app. for)

IT 7732-18-5, Water, vapor
(removal of, from water vapor-ammonia offgas from ammonium polyphosphate **manuf**., for **ammonia** recycle and decreased load on wastewater treatment facility, app. for)

IT **1310-58-3P, Potassium hydroxide**, miscellaneous 1310-65-2P, Lithium hydroxide **1310-73-2P, Sodium hydroxide**, miscellaneous
(water vapor removal with, from ammonia-water vapor offgas from ammonium polyphosphate **manuf**., for **ammonia** recycle and decreased load on wastewater treatment facility, app. for)

L56 ANSWER 7 OF 18 HCA COPYRIGHT 2003 ACS

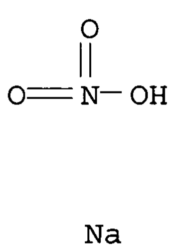
109:76252 Novel and safe explosive compositions suitable for use in underground coal mines. Seshadri, Karur Varadarajan; Sen, Gautam; Seshan, Srinivasachari; Sen, Soumendra Nath (IEL Ltd., India). Indian IN 160982 A 19870829, 40 pp. (English). CODEN: INXXAP. APPLICATION: IN 1983-CA50 19830112.

AB Permissible water-in-oil emulsion explosives comprise an aq. soln. of oxidizer salts, 2-40 wt.% nonexplosive sensitizing liquors, 1-15 wt.% flame-quenching or **coolant salts**, a d.-control agent, and, optionally, a gassing accelerator dispersed in a continuous fuel phase comprising hydrocarbon fuels and emulsifiers. The aq. soln., at 15-20.degree. above its fudge pt., is dispersed in the fuel phase at 50-80.degree. and emulsified under low shear and at a low mixing speed of 110-135 rpm. The sensitizing liquors are described in Indian Patent Applications 150613 and 157795. NH4NO3 and **NaNO3** are the oxidizer salts used in 26 formulations for which compositional data but no performance data are given. The sensitizing liquor in some of these comprises NH4NO3, hexamine dinitrate, hexamine, and water and the **coolant salts** are NaCl and KCl.

IT **7631-99-4, Sodium nitrate**, uses and miscellaneous
(explosive water-in-oil emulsions, permissible, sensitizing liquors and **coolant salts** in)

RN 7631-99-4 HCA

CN Nitric acid sodium salt (8CI, 9CI) (CA INDEX NAME)



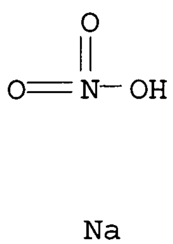
IT **7664-41-7D**, **Ammonia**, reaction **products**
with formaldehyde and nitric acid
(sensitizing liquor, in nitrate water-in-oil emulsions for
permissible explosives)
RN 7664-41-7 HCA
CN Ammonia (8CI, 9CI) (CA INDEX NAME)

NH_3

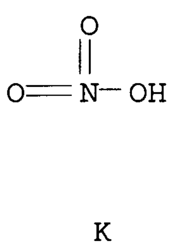
IC ICM C06B001-00
CC 50-2 (Propellants and Explosives)
IT Explosives
(emulsion, permissible, nitrate-based, contg. sensitizing liquors
and **cooling agents**)
IT Emulsions
(water-in-oil, permissible nitrate explosives of, contg.
sensitizing liquors and **cooling salts**)
IT 7447-40-7, Potassium chloride, uses and miscellaneous 7647-14-5,
Sodium chloride, uses and miscellaneous
(**cooling agents**, in nitrate water-in-oil
emulsions for permissible explosives)
IT 7732-18-5
(emulsions, water-in-oil, permissible nitrate explosives of,
contg. sensitizing liquors and **cooling salts**)
IT **7631-99-4**, **Sodium nitrate**, uses and
miscellaneous 7697-37-2, Nitric acid, uses and miscellaneous
(explosive water-in-oil emulsions, permissible, sensitizing
liquors and **coolant salts** in)
IT 50-00-0D, Formaldehyde, reaction **products** with
ammonia and nitric acid 100-97-0, uses and miscellaneous
131-73-7, Hexamine nitrate 141-43-5, Ethanolamine, uses and
miscellaneous **7664-41-7D**, **Ammonia**, reaction
products with formaldehyde and nitric acid 7697-37-2D,
Nitric acid, reaction **products** with **ammonia** and
formaldehyde 20748-72-5
(sensitizing liquor, in nitrate water-in-oil emulsions for
permissible explosives)

L56 ANSWER 8 OF 18 HCA COPYRIGHT 2003 ACS
106:216436 Manufacture of concentrated solutions of sodium, potassium,
or calcium nitrate from waste acid condensate from concentrated
nitric oxide manufacturing.. Wilk, Marcin; Kozlowski, Kazimierz
(Instytut Nawozow Sztucznych Pulawy, Pol.). Pol. PL 129987 B2
19850330, 4 pp. (Polish). CODEN: POXXA7. APPLICATION: PL
1981-234575 19811231.
AB The condensate (contg. 2-10% HNO_3), which is obtained in the prodn.
of N oxides by the **catalytic** oxidn. of NH_3, is neutralized
by scrubbing with Na, K, or Ca hydroxide or carbonate. Optionally,
the neutralization is done before scrubbing in an ion exchanger
which is regenerated by using **NaOH**, **KOH**, or
$Ca(OH)_2$. The soln. from the scrubber is concd., and the resulting

sludge (contg. .apprx.40% nitrate) is recovered.
IT **7631-99-4P**, preparation **7757-79-1P**, preparation
(manuf. of, from waste acid condensate in nitric oxide manuf.)
RN 7631-99-4 HCA
CN Nitric acid sodium salt (8CI, 9CI) (CA INDEX NAME)



RN 7757-79-1 HCA
CN Nitric acid potassium salt (8CI, 9CI) (CA INDEX NAME)



IT **7664-41-7P**, Ammonia, reactions
(oxidn. of, **catalytic**, for nitric oxides, nitrate prodn. from waste acid condensate from)
RN 7664-41-7 HCA
CN Ammonia (8CI, 9CI) (CA INDEX NAME)

NH_3

IT **1310-58-3**, reactions **1310-73-2**, reactions
(waste acid neutralization by, in nitric oxide manuf., for nitrate)
RN 1310-58-3 HCA
CN Potassium hydroxide (K(OH)) (9CI) (CA INDEX NAME)

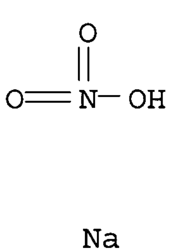
K— OH

RN 1310-73-2 HCA
CN Sodium hydroxide (Na(OH)) (9CI) (CA INDEX NAME)

Na— OH

IC C01B021-48; C01B021-26
CC 49-5 (Industrial Inorganic Chemicals)
ST **sodium nitrate** manuf waste acid condensate; **potassium nitrate** manuf acid condensate; calcium nitrate manuf acid condensate; nitric oxide waste acid condensate
IT 7697-37-2P, Nitric acid, reactions
(condensate, waste, neutralization of, with calcium hydroxide or **potassium hydroxide** or **sodium hydroxide**, in nitric oxide manuf., for nitrates)
IT 11104-93-1P, preparation
(manuf. of, by **catalytic** oxidn. of **ammonia**, waste acid **condensate** neutralization in)
IT **7631-99-4P**, preparation **7757-79-1P**, preparation
10124-37-5P, Calcium nitrate (Ca(NO3)2
(manuf. of, from waste acid condensate in nitric oxide manuf.)
IT **7664-41-7P**, Ammonia, reactions
(oxidn. of, **catalytic**, for nitric oxides, nitrate prodn. from waste acid condensate from)
IT 1305-62-0, reactions **1310-58-3**, reactions
1310-73-2, reactions
(waste acid neutralization by, in nitric oxide manuf., for nitrate)

L56 ANSWER 9 OF 18 HCA COPYRIGHT 2003 ACS
73:121242 Noncombustible, modified polyurethane foam material. Wagner, Kuno (Farbenfabriken Bayer A.-G.). Ger. Offen. DE 1911643 19700924, 32 pp. (German). CODEN: GWXXBX. APPLICATION: DE 1969-1911643 19690307.
AB Open-celled polyurethane foams are impregnated with a soln. of urea, a high-mol.-wt. .alpha.,.omega.-diurea or diurethane (prepd. from OCN(CH2)6NCO and a polyethylene glycol), aq. HCHO, and HCO2H **catalyst** or a similar soln. and heated to form an aminoplast resin in the foam, giving a foam having high tensile strength, elongation, and elasticity and fire resistance.
IT **7631-99-4**, uses and miscellaneous
(urethane polymer foams contg.)
RN 7631-99-4 HCA
CN Nitric acid sodium salt (8CI, 9CI) (CA INDEX NAME)



IC C08G
CC 36 (Plastics Manufacture and Processing)

IT Urea condensation products, preparation
Urea **condensation products**, with **ammonia**
, formaldehyde, hexamethylene isocyanate and polyethylene glycol, preparation
Ammonia, polymer with formaldehyde, hexamethylene isocyanate, polyethylene glycol and urea
Glycols, polyethylene, polymer with ammonia, formaldehyde, hexamethylene isocyanate and urea
Isocyanic acid, hexamethylene ester, polymer with ammonia, formaldehyde, polyethylene glycol and urea
(manuf. of, in urethane polymer foams)

IT 7447-40-7, uses and miscellaneous 7487-88-9, uses and miscellaneous **7631-99-4**, uses and miscellaneous 7757-93-9 7778-80-5, uses and miscellaneous 7783-20-2, uses and miscellaneous 13011-54-6
(urethane polymer foams contg.)

L56 ANSWER 10 OF 18 HCA COPYRIGHT 2003 ACS

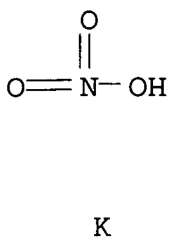
63:28023 Original Reference No. 63:4988a-b Development of a method for **producing** an iron, **ammonia synthesis** catalyst with four promotors. Lipinskaya, V. P. (Nitrogen-Fertilizer Plant, Kemerovo). Nauchn. Osnovy Podbora i Proizv. Katalizatorov, Akad. Nauk SSSR, Sibirsk Otd. 109-13 (Russian) 1964.

AB A method is described for prepg. an Fe catalyst for **NH3 synthesis** contg. 4 promotors. The catalyst is prepd. by oxidizing fusion in a stream of O in 2 steps: (1) 9 kg. of Fe is fused in a stream of O, and (2) oxidized in the O until the FeO concn. is 30-35%. The promotors are added in the following order with a new portion of the Fe (9 kg.): 0.5 kg. SiO2, 1.5 kg. Al2O3, 2.2 kg. CaO, 0.8 kg. **KNO3**. When the last portion of the Fe is oxidized the flow of O is discontinued and the melt is poured into a **cooling** vat. The temp. of fusion of the catalyst is 1600-1700.degree.. After hardening the product is ground and fused in an elec. furnace with the addn. of 3-4 kg. Fe/300 kg. of ground catalyst to obtain the desired FeO concn. The melt is rapidly passed through a tap hole into a **cooling** vat **cooled** by H2O **flowing** through its jacket. The **cooled** catalyst is ground and sorted as to grain size into 5 fractions: 0-2, 2-4, 4-6, 6-8, and 8-12 mm.

IT **7757-79-1**, **Potassium nitrate**
(catalysts from Fe, oxides and, for **NH3 manuf**
.)

RN 7757-79-1 HCA

CN Nitric acid potassium salt (8CI, 9CI) (CA INDEX NAME)



CC 5 (Catalysis and Reaction Kinetics)
IT Lime
(catalysts from Fe, oxides, **KNO3** and, for **NH3 manuf**.)
IT Catalysts and Catalysis
(for **ammonia manuf**., Fe as)
IT Promoters
(for iron catalyst, in **NH3 manuf**., oxides and **KNO3** as)
IT **Ammonia**, **manufacture** of
(catalysts for, four promoters in)
IT **7757-79-1**, **Potassium nitrate**
(catalysts from Fe, oxides and, for **NH3 manuf**.)
IT 7439-89-6, Iron
(catalysts, for **NH3 manuf**.)

L56 ANSWER 11 OF 18 HCA COPYRIGHT 2003 ACS
58:33017 Original Reference No. 58:5547e-h Synthesis of sugar-reducing derivatives of phenylsulfonylurea. Makhnenko, N. I.; Sysoeva, T. F. Tr. Ukr. Inst. Eksperim. Endokrinol, 18, 333-5 From: Ref. Zh., Khim. 1962, Abstr. No. 12Zh160. (Unavailable) 1961.
AB In the search for effective prepns. for treatment of sugar diabetes p-RC6H4SO2NHCONHR' (I) (Ia-d, where R = Me, R' = C6H11; R = Cl, R' = C6H11; R = Cl, R' = Bu; R = Cl, R' = Pr) were synthesized by previously described methods. Ia and Ib were obtained by the reaction of urethan with an amine; Ic and Id were obtained-by the reaction of BuNCO with p-ClC6H4SO2NH2 (II). p-MeC6H4SO2NH2, (186.1 g.), 102.6 g. urea, 67 g. **KOH**, and 335 g. PhCH2OH was heated until gas evolution ceased. The mixt. was filtered and the filtrate acidified with dil. HCl to give I (R = Me, R' = H) (III), yield 83.4%, m. 185-7.degree.. Id (233 g.) in 1130 ml. MeOH was heated 1 hr. to boiling, after 20 hrs. 32.6 ml. concd. H2SO4 added, and after 2 addnl. hrs., the mixt. was filtered. The filtrate was treated with Na2CO3, the mixt. filtered, and the filtrate acidified with dil. HCl to give p-MeC6H4SO2NMeCO2Et (IV), yield 79.6%, m. 105-7.degree.. PhOH (300 ml.), 204 ml. concd. NH4OH, and 12 g. Raney Ni at 195-200.degree. was heated 14 hrs. in an autoclave, the water layer removed, and concd. HCl added. The acid soln. was evapd. to dryness, the amine salt decompd. with 20% alkali, and C6H11NH2 (V), yield 29.8%, b. 134.degree., distd. V was also

obtained in 41% yield by redn. of cyclohexanone oxime. A mixt. of 100.7 g. IV and 338.7 ml. anhyd. PhMe was heated in a sand bath, moisture evapd., 41.2 g. V added after cooling, heated 10 hrs. on a water bath, the reaction product extd. with 5% alkali, the alk. soln. heated (50.degree., 3 hrs.) to remove PhMe, **cooled**, the **salt** filtered off, and the mixt. acidified with dil. HCl (1:1) to give 56.3% Ia, m. 171-3.degree. (alc.). Ib was obtained similarly. p-ClC6H4SO2Cl reacted with **NH3** to **form** 51% II, m. 143.degree.. II with NH2CONH2 in the presence of an alkali in C6H11OH yielded p-ClC6H4SO2NHCONH2, yield 85.8%, m. 126-8.degree.. This compd. forms the corresponding urethan in MeOH in the presence of concd. H2SO4. This urethan reacts with V to form 26-30% Ib, m. 184-5.degree.. II with BuNCO yielded 49.4% Ic, m. 115-16.degree.. Id was obtained in the same way in 37.4% yield, m. 128-9.degree.. Ia and Id were effective for oral treatment of sugar diabetes.

CC 35 (Noncondensed Aromatic Compounds)

L56 ANSWER 12 OF 18 HCA COPYRIGHT 2003 ACS

57:36212 Original Reference No. 57:7202e-i,7203a-f Amines derived from dihalopropenes. II. Synthesis of (.+-.)- and (-)-1-(2-methylene-1-aziridinyl)-3-buten-2-ol. Bottini, Albert T.; Dev, Vasu (Univ. of California, Davis). Journal of Organic Chemistry, 27, 968-79 (Unavailable) 1962. CODEN: JOCEAH. ISSN: 0022-3263.

AB cf. CA 56, 14037b. Treatment of H2C:CBrCH2NHCH2CH(OH)CH:CH2 (I) with NaNH2 in liquid NH3 gave the title compd. (II) together with a small amt. of HC:CCH2NHCH2CH(OH)CH:CH2 (III). Hexamethylenetetramine (154 g.) in 1250 ml. CHCl3 refluxed 1 hr. with dropwise addn. of 200 g. CH2:CBrCH2Br, the mixt. refluxed 4 hrs., kept 16 hrs., and **cooled** (ice bath) gave 308 g. air-dried quaternary ammonium bromide, m. 185.0-6.5.degree.. The salt (200 g.) in 2400 ml. warm 5:1 alc.-H2O and 480 ml. concd. HCl kept 2 days, the filtrate evapd., the residue taken up in 250 ml. H2O, 7 made alk. with 6N **NaOH**, the aq. layer satd. with NaCl, extd. with Et2O, the combined org. phases washed (satd. aq. NaCl), dried (K2CO3), and evapd. gave 72% toxic H2C:CBrCH2NH2 (IV), b100 65-7.degree., n3D5 1.5081; p-bromobenzenesulfonamide deriv. m. 90-2.degree. (use of gloves and goggles in prepn. and handling of IV advised). (CH2CO)2NBr (2.5 moles) in 2.8 moles H2C:CHCH:CH2 and 50 ml. Et2O stirred vigorously 6 hrs. with 500 ml. 2:3 Et2O-H2O at 10.degree., the aq. phase extd. with Et2O, the combined Et2O solns. dried (MgSO4), and the residue on evapn. distd. (N atm.) through a glass helices-packed column yielded 81% H2C:CHCH(OH)CH2Br (V), b6 54-5.degree., n2D2 1.5013. V (5.68 moles) added in 5 hrs. with stirring to 34.2 moles **NaOH** in 27 l. H2O at 16-18.degree. and the mixt. stirred 3 hrs., the aq. layer washed with Et2O, and the combined dried Et2O solns. distd. yielded butadiene monoxide (VI), b. 65.5-7.0.degree., n2D6 1.4129. VI (0.15 mole) added dropwise in 20 min. with stirring to 0.46 mole IV and 2 ml. H2O at 15.degree., the mixt. heated slowly to 100.degree., the temp. maintained 6 hrs., and unchanged IV (41 g.) removed at 52-4.degree./30 mm. gave 74% residual I, b2 107-9.degree., n2D6.5

1.5128. NaNH2 (0.105 mole) in 200 ml. liquid NH3 mechanically stirred 30 mln. with dropwise addn. of 0.050 mole I in 30 ml. dry Et2O, the mixt. refluxed (solid CO2 **cooled condenser**) 6 hrs., the **NH3** evapd., the stirred residue dild. with 50 ml. H2O, the soln. satd. with NaCl, extd. with Et2O, the exts. washed with satd. aq. NaCl, the dried (K2CO3) ext. evapd., and the residue distd. (N atm.) yielded 4.1 g. distillate, b1.0 50-68.degree., n2D5 1.4840, v 2100, 1785 cm.-1, taken up in 40 ml. dry Et2O and **cooled** to -10.degree. gave 0.9 g. essentially pure III, m. 59-60.degree.. The mother liquor concd. and **cooled** to -25.degree. gave 0.2 g. mixt. of III and II. The filtrate dried over K2CO3 and distd. gave 2.5 g. II, b1.5 50-2.degree. n3D3.5 1.4827. Another run in which 0.10 mole I was treated with 0.21 mole NaNH2 and the products fractionated (N atm.) yielded 6.4 g. 98% pure II, b3 65-8.degree., n2D9 1.4845, together with 2.0 g. material, b3 68-86.degree., n2D9 1.4824, contg. mainly III. Abs. alc. (300 ml.) contg. 0.17 mole l-1-amino-3-buten-2-ol d-cam-phor-10-sulfonate [m. 147.5-8.5.degree., [.alpha.]27 14.4.degree. (0.160 g./10 ml. H2O), Ettlinger, CA 45, 3381f] treated with 0.17 mole **KOH** in 150 ml. abs. alc., the filtered soln. concd. at 50 mm., the residue dild. with 50 ml. alc., and the filtered soln. distd. yielded 64% (-)-H2C:CHCH(OH)CH2NH2 (VII), m. 52.3.degree., b10 77-9.degree., [.alpha.]22D -28.0.degree. (0.199 g./10 ml. H2O). VII (0.10 mole) and 0.050 mole H2C:CBrCH2Br refluxed 4 hrs. in 75 ml. abs. alc., the mixt. distd. at 50 mm. to a pot temp. of 55.degree., the **cooled** residual oil dild. with 100 ml. Et2O, filtered, the residue washed with Et2O, and the combined filtrate and washings distd. yielded 63% (-)-I, m. 34-6.degree., [.alpha.]23D -3.5.degree. (0.263 g./10 ml. abs. alc.). NaNH2 (2.05 g.) and 0.025 mole (-)-I **condensed** in liquid **NH3** and the **product** distd. (N atm.) yielded 31% (-)-II, b1-5 51-3.degree., n21D 1.4878, [.alpha.]20D -30.5.degree. (0.0837 g./20 ml. abs. alc.), contg. less than 2% active III, together with 1.05 g. 2nd fraction, b1-5 54-67.degree., refractionated to give 0.4 g. (-)-II. VI (0.2 mole) and 144 ml. 25% aq. HNMe2 (0.8 mole) refluxed 6 hrs. (solid CO2 **cooled** condenser), the **cooled** (ice bath) mixt. made strongly alk. with 50 g. **NaOH**, the aq. layer extd. with Et2O, and the dried (K2CO3) org. solns. distd. yielded 61% H2C:CHCH(OH)CH2NMe2, b33 66-7.degree., n23D 1.4472; MeI salt m. 70-9.degree.. Several attempts were made to convert the corresponding hydroxide to VI, since H2C:CHCH(OH)CH2NH2, which had been resolved, could be readily converted to the hydroxide. The only N-free compds. isolated were MeOH and Me-COCH:CH2. The structure of II was confirmed by comparison of its n.m.r. spectrum with that of 1-ethyl-2-methyleneaziridine and of H2C:CHCH(OH)CH2NEt2. II appeared to have greater antineoplastic activity than (-)-II against the mouse tumor Adenocarcinoma 755 and 4 times less activity than the broad spectrum antineoplastic agent tetramine.

CC 31 (Heterocyclic Compounds-One Hetero Atom)

L56 ANSWER 13 OF 18 HCA

54:67962 Original Reference No. 54:13022d-i,13023a-f Synthesis of cyclohexylideneacetaldehyde and 2-, 3-, and 4-methylcyclohexylideneacetaldehyde. Chaco, M. C.; Iyer, B. H. (Indian Inst. Sci., Bangalore). Journal of Organic Chemistry, 25, 186-90 (Unavailable) 1960. CODEN: JOCEAH. ISSN: 0022-3263.

AB Cyclohexanone (I) and 2- (II), 3- (III), and 4-methylcyclohexanones (IV) were condensed with C2H2 (V) to give the resp. 1-ethynylcyclohexanols (VI) (VII) (VIII) (IX). VI-IX were hydrogenated to the resp. 1-vinyl- and 1-ethylcyclohexanols. The 1-vinylcyclohexanols treated with PBr3 gave the corresponding rearranged .beta.-cyclohexylideneethyl bromides, which were converted to the pyridinium salts. The latter were treated with p-nitrosodimethylaniline (X) and alkali to give the corresponding nitrones, which were hydrolyzed to the corresponding aldehydes. The ethynyl, vinyl, and 1-ethylcyclohexanols were tested pharmacol. I-IV were condensed with V in liquid NH3 in the presence of C2HNa. I (1 mole) in 150 cc. Et2O was added during 30 min. to C2HNa (from 1.1 mole Na with Fe(NO3)3 in 1 l. NH3), a continuous stream of V introduced, the passage continued 4 hrs., a further 500 cc. liquid NH3 added, the mixt. kept overnight, the **NH3** evapd., the **product** worked up with H2O and Et2O, the Et2O washed, dried, and distd. The following results were obtained (compd. formed, b.p./mm., and % yield given): VI, 86-8.degree./26, 75.2; VII, 98-102.degree./44, 92; VIII, 79-82.degree./10, 76; IX, 76-80.degree./10, 76.5. VII, VIII, and IX were **chilled** by ice-**salt** mixt., filtered through a cold funnel, and the residues recrystd. from ligroine. Redistn. of the filtrates gave the liquid forms. The following m.ps. and 3,5-dinitrobenzoates were obtained (compd., m.p., and m.p. of the 3,5-dinitrobenzoate given): VII, 60.degree., 101.degree.; VIII, 23-5.degree., 113-15.degree.; IX, 43.degree., 147-8.degree.. V (30 g.) shaken with H over Pd-CaCO3 in 160 cc. alc., the catalyst removed, and the residue distd. gave 26 g. 1-vinylcyclohexanol (XI). In a similar manner the 2- (XII), and 3-methyl-1-vinylcyclohexanols (XIII) were prepd. from VII and VIII. For the prepn. of 4-methyl-1-vinylcyclohexanol (XIV), Pd-PbCaCO3 catalysts were used in the presence of quinoline until the calcd. vol. of H was absorbed, the catalyst removed, solvent evapd., and the residue distd. to give 17 g. liquid XIV. The following results were obtained (product, b.p./mm., m.p. of 3,5-dinitrobenzoate given): XI, 77-80.degree./25, -; XII (solid), 49.degree./0.5-1.0, 104-5.degree.; XII (liquid), 72-4.degree./11.5, -; XIII (solid), 51.degree./1, 91-2.degree.; XIII (liquid), 61.degree./3, -; XIV (solid), 54.degree./1.5, 111.degree.; XIV (liquid), 49.degree./0.5-1.0, -. VI (4 g.) shaken with H in the presence of 0.25 g. PdCaCO3 in alc. until H absorption ceased, the catalyst removed, the solvent evapd., and the residue distd. gave 3.25 g. 1-ethylcyclohexanol (XV). Similarly, 2- (XVI), 3(XVII), and 4-ethylcyclohexanols (XVIII) were prepd. (compd., m.p., b.p./mm., m.p. of 3,5-dinitrobenzoate given): XV, -, 81.5.degree./2, -; XVI (solid), -, 94.degree./23, 138.degree.; XVI (liquid), -, 91-2.degree./24.5, -; XVII (solid), -, 66.degree./3.5, 91-2.degree.; XVII (liquid), -, 83-5.degree./15, -; XVIII (solid), 30.degree.,

64.degree./3, 114.5.degree.; XVIII (liquid), -, 53.degree./1.5-2.0, -. XI (16 g.) contg. 1 g. anhyd. C5H5N in 30 cc. ligroine treated slowly with 20 g. PBr3, after 16 hrs. at room temp. the mixt. decompd. with cold H2O, extd. with ligroine, the ext. washed free of acid, dried, and evapd. gave 15 g. .beta.-cyclohexylideneethylene bromide (XIX), b11 78-83.degree.. The following were similarly prepd. (compd., % yield, and b.p./mm given): 2-methyl-.beta.-cyclohexylideneethyl bromide (XX), 72, 70-5.degree./2.5; 3-methyl-.beta.-cyclohexylideneethyl bromide (XXI), 77.6, 70-5.degree./4; 4-methyl-.beta.-cyclohexylideneethyl bromide (XXII), 68.6, 66-72.degree./3.5. XIX (7.9 g.) mixed with 38 cc. anhyd. C5H5N and kept well stoppered 24 hrs., the excess C5H5N removed, and the residue washed with ligroine gave 8.78 g. cyclohexylideneethylpyridinium bromide (XXIII). In a similar manner, XX gave 76.5% 2-methyl-.beta.-cyclohexylideneethylpyridinium bromide (XXIV); XXI gave 92.4% 3-methyl-.beta.-cyclohexylideneethylpyridinium bromide (XXV), m. 80-5.degree.; XXII gave 86.5% 4-methyl-.beta.-cyclohexylideneethylpyridinium bromide (XXVI), m. 60-2.degree.. XXIV (8.9 g.) in 15 cc. MeOH treated at 0.degree. with 6.2 g. X in 80 cc. MeOH, the mixt. shaken and refrigerated 16 hrs. with 37 cc. N **NaOH**, 160 cc. cold H2O added, the mixt. shaken and chilled 24 hrs. longer, and the solid collected gave 2.8 g. 2-methylcyclohexylidenemethyl-N-(p-dimethylaminophenyl)nitrone, yellow needles, m. 67-8.degree. (ligroine-EtOAc). When similar expts. were carried out with XXIII, XXV, and XXVI, the nitrones sepd. as dark smeary liquids which did not solidify. XXIII (8.7 g.) in 15 cc. MeO cooled to 0.degree., treated with 10 g. X in 20 cc. MeOH and 120 cc. C6H6, 35 cc. **NaOH** added, the mixt. refrigerated 40 hrs., dild. with H2O, the aq. layer sepd., extd. with more C6H6, the exts. washed with 2N HCl, then with NaHCO3, dried, evapd., and the residue distd. gave 1.9 g. cyclohexylideneacetaldehyde, b11.5 88.degree., n32D 1.5032, .lambda. 241 m.mu., .epsilon. 16,810; semicarbazone m. 210.degree., .lambda. 273 m.mu., .epsilon. 32,200; 2,4-dinitrophenylhydrazone, red plates, m. 211-2.degree., .lambda. 256, 290, 386 m.mu., .epsilon. 16,600, 9843, 27,450. The following results were similarly obtained. XXIV gave 48.8% 2-methylcyclohexylideneacetaldehyde, b3 78-80.degree., n25.5D 1.4985, .lambda. 241 m.mu. .epsilon. 17,270 (semicarbazone m. 204.degree., .lambda. 273 m.mu., .epsilon. 32,500; 2,4-dinitrophenylhydrazone, orange yellow plates, m. 167-9.degree., .lambda. 256, 290, 386 m.mu., .epsilon. 16,530, 10,600, 28,490); XXV gave 46.8% 3-methylcyclohexylideneacetaldehyde, b4 75-7.degree., n25D 1.4990, .lambda. 241 m.mu., .epsilon. 18,800 (semicarbazone m. 192-3.degree., .lambda. 273 m.mu., .epsilon. 32,070; 2,4-dinitrophenylhydrazone, orange red needles, m. 158-9.degree., .lambda. 256, 290, 386 m.mu., .epsilon. 16,290, 10,140, 28,030); XXVI gave 50.2% 4-methylcyclohexylideneacetaldehyde, b1 68-70.degree., n25D 1.4985, .lambda. 240 m.mu., .epsilon. 18,640 (semicarbazone m. 198-200.degree., .lambda. 273 m.mu., .epsilon. 31,670; 2,4-dinitrophenylhydrazone, orange plates, m. 177-8.degree., .lambda. 256, 290, and 386 m.mu., .epsilon. 17,590, 10,480, 28,180).

CC 10D (Organic Chemistry: Alicyclic Compounds)

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51:85749 Original Reference No. 51:15533f-i,15534a-i,15535a-g
Quebrachamine. I. Witkop, Bernhard (Natl. Insts. of Health, Bethesda, MD). J. Am. Chem. Soc., 79, 3193-200 (Unavailable) 1957. CODEN: JACSAT. ISSN: 0002-7863.

AB Quebrachamine (I) (50 mg.), m. 147.degree. intimately mixed with 7 g. Zn dust and dry distd., 50 such distns. carried out, the combined Et2O solns. of the distillates concd. to 50 cc. and extd. with dil. HCl, the acidic ext. basified and extd. with Et2O, the ext. evapd., the residue steam-distd., the distillate salted with NH4Cl and extd. with Et2O, the ext. evapd., the few drops of colorless oily residue dissolved in 2 cc. dil. HCl, the soln. treated with excess aq. picric acid, the flocculent ppt. filtered off after 1 hr. and washed with H2O, the dry residue triturated with 0.5 cc. cold Me2CO, and the cryst. material recrystd. from 1 cc. Me2CO gave 3,5-diethylpyridine picrate, fine glistening needles, m. 180-1.degree. (all m.ps. are cor.). The basic residue from the steam distn. dissolved in Et2O and slowly evapd. gave unchanged I, m. 144.degree.. The HCl-extd. Et2O soln. evapd., the residue steam-distd., the volatile portion distd. in vacuo, the distillate dissolved in petr. ether, and the soln. treated with excess picric acid in warm petr. ether (b. 60.degree.) gave a ppt. which recrystd. 7 times from C6H6-petr. ether gave the mixed isomorphous picrates of .beta.-methyl- and .beta.-ethylindole. The dark residue from the steam distn. of the indoles distd. at 140.degree. and 0.01 mm. and the colorless cryst. distillate recrystd. from C6H6 gave sheaves of fine needles, m. 218.degree., probably a mixt. of carbazole and its 2(or 3)-Me homolog; the Ehrlich reaction gave a light red color in the cold, dark red on warming, fading on **cooling**, and changing to dark blue-violet with a trace of KNO2. I (0.5 g.) and 0.5 g. Pd black heated 4 hrs. at 320.degree. (NH3 was evolved), the mixt. treated with Et2O and filtered, the filtrate extd. with dil. HCl, the ext. basified and extd. with Et2O, the final ext. evapd., the brown semisolid residue heated at 100.degree./1 mm. and distd. at 180.degree./0.01 mm., the resulting droplet of a light yellow oil (solidifying in the cold) dissolved in 0.1N HCl and converted to the picrate, the dry picrate triturated with 0.2 cc. glacial AcOH, and the yellow residue washed with Et2O and recrystd. from Me2CO yielded 1% clusters of needles, m. 257.degree. (decompn.), which slowly crystd. from more dil. Me2CO soln. gave short prisms; this material is the picrate of a base, C14H16N2 (II). II picrate in Me2CO acidified with 4N HCl and extd. with Et2O, and the blue fluorescing aq. layer worked up gave II.HCl, sheaves of needles, m. 210.degree. (from EtOH-Et2O). II.HCl treated with aq. alkali and the base isolated with Et2O gave II, needles, m. 202.degree.. I dehydrogenated with Se at 300.degree. gave **NH3** volatile **products**, and a neutral compd. which in C6H6 after filtration through Al2O3 showed a strong green fluorescence; dehydrogenation with S gave NH3 and H2S beginning at 150.degree.; the amorphous reaction products seemed to contain in both cases Se

or S, resp. I (1 g.) in 10 cc. 60% AcOH treated 2 hrs. with **cooling** with a **stream** of 2.5% ozone in O, the mixt. adjusted to pH 8, the light brown ppt. (0.4 g.) continuously extd. with Et2O, and the ext. slowly evapd. gave a base, C21H28N2O2 (III), prisms, which recrystd. from Me2CO gave needles, m. 213.degree.; microsublimation of the viscous mother liquor gave a 2nd crop of III. I (0.5 g.), 3 cc. glacial AcOH, and 3 cc. 30% H2O2 kept 40 hrs. at 20.degree., **cooled**, adjusted with alkali to pH 7.5-8.0, the pptd. pink powder dissolved in Me2CO, and the soln. slowly concd. gave III, long glistening needles, m. 213.degree.; it sublimed in vacuo at 180-200.degree.. Brief treatment of III with 2N mineral acid seemed to form a diazotizable amine while longer treatment with 4N HCl gave a new base, C19H26N2O (IV), m. 103.degree.. III (0.004M) in 60% aq. EtOH at 20.degree. had a pH of 9.1; pK 7.85. III (80 mg.) in Et2O reduced with excess LiAlH4 gave a base (IVa), m. 132-4.5.degree., which treated in Et2O with dry HCl gave V.HCl, hygroscopic powder, m. 155-65.degree.. III (50 mg.) in 4 cc. 4N HCl kept 3 hrs. on the steam bath, and evapd. to dryness in a desiccator at room temp., the residue treated with aq. alkali, and the ppt. recrystd. from Et2O gave IV, fine needles, m. 103.degree.. I (1 g.) ozonized, the resulting III pptd. at pH 8, and the filtrate basified strongly with excess 50% **NaOH** and extd. with Et2O gave on slow evapn. of the Et2O the base, C19H26N2O (V), prisms, m. 188.degree.. I (1 g.) in 15 cc. MeOH treated with **cooling** with excess 3% ozone in O and the mixt. evapd. gave 1.3 g. light brown powder which was readily sol. in alkali and did not blue KI-starch paper. I (184 mg.) in CHCl3 left 24 hrs. with 140 mg. BzO2H in CHCl3, the red soln. washed with dil. alkali and evapd., the residue dissolved in 2N HCl, the soln. adjusted with alkali to pH 8, washed with Et2O, made strongly alk. and extd. with Et2O, and the ext. evapd. gave 110 mg. V, m. 186-8.degree. (from Et2O). The filtrate (pH 8) from the prepn. of III made strongly alk. and extd. with Et2O, and the ext. worked up gave 450 mg. V, large prisms, m. 188.degree. (from Et2O). V treated 24 hrs. at room temp. with excess Ac2O was recovered unchanged. Slow recrystn. of V from Me2CO gave an Me2CO adduct, clusters of needles, m. 115.degree., which dried 2 days at 100.degree. in vacuo yielded V, m. 178-80.degree.. A series of runs with various reaction times were carried out for the oxidation of 500 mg. I with a mixt. of 3 cc. each of glacial AcOH and 30% H2O2 at 20.degree. (reaction time in hrs., mg. recovered I, mg. V, mg. III, and % acidic products not pptd. by base given): 14, 200, 290, -, -; 24, -, 470, 35, -; 40, -, 450, 45, little; 100, -, -, -, above 90. Rapid recrystn. of V from Me2CO gave pure V, m. 186-8.degree., [.alpha.]D20 504.degree. (c 1.0, 50% aq. EtOH). I gives a lasting strong, purple Hopkins-Cole test; V gives similarly a red color fading to blue after 20 hrs. V gave a pink Ehrlich test turning red on warming. V (0.01M) in H2O at 20.degree. had pH 11.36; titration with 0.01N HCl gave pK 10.57 and 7.43 (on back-titration with 0.01N **NaOH** a marked hysteresis was observed indicating rearrangement during the titration). pK in 80% Methyl Cellosolve: I 6.76, V 11.9 and 5.65. V in Et2O treated with dry HCl gave

V.HCl.0.75H2O, prisms, m. 285-7.degree. (sublimed at 200-40.degree.). V.HCl.0.75H2O in H2O treated with aq. picric acid gave V picrate, yellow scales, m. 173.degree.. V in Et2O treated with excess MeI and the cryst. ppt. recrystd. from MeOH-Et2O gave V.HI, needles, m. 280.degree. with darkening at 260.degree. and sintering at 275.degree. (decompn.), which reduced with LiAlH4 or NaBH4 gave I. V (200 mg.) in Et2O reduced with LiAlH4 gave I, m. 145-7.degree., 60 mg. oily, Et2O-sol. base [picrate yellow prisms, m. 170-2.degree. (from MeOH)], and 10 mg. flocculent Et2O-insol. product which crystd. from CHCl3 yielded short prisms, m. 183-6.degree.. I was recovered unchanged after refluxing 1 hr. with excess LiAlH4 in tetrahydrofuran. V (0.3 g.) in 4 cc. glacial AcOH hydrogenated over 100 mg. PtO2 until 2 equivs. H had been absorbed, and filtered, the filtrate adjusted to pH 8, and the flocculent ppt. (about 40 mg.) crystd. from Me2CO gave the hexahydro-deriv. (VI) of V, prisms, m. 177.degree.; the aq. mixt. dild. further with 50% aq. alkali pptd. 2 addnl. basic fractions at pH 12 and 14; the latter ppt. crystd. overnight at 0.degree. from Me2CO gave the dihydro deriv. (VII) of V, cushions of needles, m. 136-8.degree.; it gave a wine-red Ehrlich color test. VII hydrogenated further and worked up at pH 8 gave VI, m. 175.degree.; further processing at pH 14 gave VII.Me2CO, m. 141-5.degree., resolidified at 165.degree. and rem. 175.degree.. Equal parts V and Pd black heated to 300.degree. gave no NH3 odor. VI (500 mg.) and 300 mg. Pd black heated 2 hrs. to 300-20.degree. evolved 120 cc. H, the mixt. extd. with Et2O, the residue from the ext. distd. at 150.degree./14 mm., the oily basic distillate (104 mg.) extd. into 0.1N HCl, and the ext. treated with aq. picric acid gave a picrate of C11H17N, yellow needles, m. 172-4.degree. (from Me2CO), undepressed with picrate A, m. 168-72.degree., from aspidospermine. V (0.3 g.) and 1 g. **KOH** in 5 cc. AmOH refluxed 1 hr. and evapd. to dryness in vacuo, the residue sepd. into acid, basic, and neutral fractions, and the Et2O-sol. basic fraction (120 mg.) chromatographed on Al2O3 with 1:1 CHCl3-C6H6 gave a cryst. indoxyl fraction, C19H26N2O2.1.5H2O (VIIa), m. 142.degree. (from Et2O); further elution with pure CHCl3 and the crude product triturated with Et2O gave an oxindole fraction, C19H26N2O2.H2O (VIII), m. 295-315.degree. (decompn.). When in the prepn. of V (2 g.) the product was left in contact overnight with strong base a small amt. of Et2O-insol. material was formed; it formed on slow evapn. from Me2CO buttons of birefringent needles (IX) which showed a cryst. transformation to slim rods at 212-40.degree., sublimed at 273.degree., and m. 278-81.degree. (decompn.). I (0.3 g.) in 5 cc. glacial AcOH and Pt (obtained by reducing 100 mg. PtO2 in 3 cc. AcOH) shaken 1.5 hrs. under O, filtered, adjusted to pH 9 with 4N **KOH** at 0.degree. filtered, treated with concd. alkali at 0.degree., and extd. with Et2O, and the ext. worked up gave base, C19H26N2O2 (X), short needles, m. 183.degree. (yellow melt, evolution of gas), it turned starch-KI paper blue. X (0.01M) in 50% aq. EtOH at 20.degree. had pH 12.3, p/K 12.13 and 9.65. X in 80% Methyl Cellosolve gave pK inflection points at 11.0 and 5.5 on titration with 0.01N HCl. X in CHCl3 treated with dry HCl gave X.HCl.H2O,

tufts of needles, m. 180-5.degree. subliming in needles at 210.degree. and rem. 275-8.degree.. A small sample of X heated to 180.degree./0.001 mm. and the cryst. material on the cold finger crystd. from Et2O gave V, prisms, m. 188.degree. (from Et2O) (picrate, m. 172.degree.). X (0.5 g.) in 40 cc. CHCl3 contg. a few drops 4N HCl in Et2O refluxed 50 min. and **cooled** deposited V.HCl.3/4H2O, m. 285-7.degree. (sublimed at 200-40.degree.); the mother liquors chromatographed on 20 g. Al2O3 gave 24 mg. Et2O-insol. HCl salt (XI). The infrared absorption spectra of I, III, IV, V, VI, and X are recorded. The infrared absorption max. in CHCl3 are listed for II, IVa, V, V.HCl.3/4H2O, VI, VIIa, VIII, IX, X, and XI, and the ultraviolet absorption max. in 95% EtOH for III, IVa, V, V.HI, VIIa, and VIII.

CC 10 (Organic Chemistry)

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49:15976 Original Reference No. 49:3137a-i,3138a-i,3139a-i,3140a-i,3141a-i,3142a-i,3143a-i,3144a-i,3145a-i,3146a-i,3147a-i,3148a-i,3149a-i,3150a-i,3151a-b Oxazoles and oxazolones. Cornforth, J. W.; Clarke, H. T.; et al. (Oxford Univ.;Princeton Univ. Press). Chemistry of Penicillin 688-848 (Unavailable) 1949.

GI For diagram(s), see printed CA Issue.

AB OXAZOLE SECTION: New methods for constructing the oxazole ring have been devised and the behavior of functional groups elucidated.

L56 ANSWER 16 OF 18 HCA COPYRIGHT 2003 ACS

21:25000 Original Reference No. 21:3048c-i,3049a-g Formation of arylnitrosohydroxylamines. Bigiavi, Dino; Franceschi, Franco Gazz. chim. ital., 57, 362-83 (Unavailable) 1927.

AB PhN(:O):NC6H4NO2-p (1.2 g.) added at 0.degree. to a soln. prepd. by filtering H3ClNOH (0.7g.) and alc. KOEt (0.8 g. K in 25 cc. abs. EtOH), let stand 24 hrs., filtered, the residue ground in water, filtered, washed, the filtrate cooled to 0.degree., satd. with Et2O, acidified with dil. H2SO4, the ppt. extd. with Et2O, and the ext. evapd. in vacuo, gives p-nitro-.alpha.-azoxybenzenenitrosohydroxylamine PhN(:):NC6H4N(:O):NOH, orange, m. 90-2.degree. (decompn.); its soln. in hot dil. **KOH** ppts. on **cooling** the K **salt**, orange. This in turn gives in water with Co(NO3)2 the Co salt, orange-yellow, insol. in dil. HCl; with Ni(OAc)2 the Ni salt, light maroon-red; with ZnCl2. the Zn salt, orange-red; with FeCl3 the Fe salt, dark maroon-red; and with HgCl2 the Hg salt, yellowish, gelatinous. In the same way is prepd. p-nitro-.beta.-azoxybenzenehydroxylamine, PhN:N(:O)C6H4N(:O):NOH, (I), orange, m. 97-8.degree. (decompn.), ppts. in Et2O with PhHNNH2 a phenylhydrazine salt, pale orange, m. 117.degree. (decompn.), and in Et2O with alc. NH2OH ppts. a hydroxylamine salt, bright yellow, turns brown around 160.degree. and m. 200-4.degree. (decompn.). From the aq. K salt of I and an aq. soln. of the particular salt are prepd. the following salts of I:Co, orange-yellow; Ni, greenish gray; cupric, grayish; Bi, orange; Cd, orange, Zn, gelatinous orange; Pb, chrome-yellow; ferric, maroon-red; mercuric, gelatinous; Al, orange-red. The K salt (loc. cit.), lustrous yellow, turns

brown at 245.degree., becomes very dark at 260.degree. and at higher temps. decomps. The NH4 salt, **prepd**. by bubbling **NH3** through I in anhyd. Et2O, is yellow and melts at a high temp. to a black liquid with evolution of reddish vapors. p-ONC6H4NO2 (cf. Ber. 36,3808(1903)) (1 g.) in hot EtOH (25 cc.) dild. with water (50 cc.), when cold the HON:NO2Na (II) (0.9 g.) slowly added with vigorous agitation, heated 10 min. at 80.degree., the p,p'-dinitroazoxybcnzene sepd, the filtrate distd. in vacuo and the salt recrystd. from water, gives Na p-nitrophenylnitrosohydroxylamine, p-O2NC6H4N(:O):NONa (III), pomegranate color, but red-yellow in powder form, becoming bright yellow at 140.degree., darkening at 200.degree., blackening at 260.degree. and then fusing to a brown liquid. It contains 5 mols. of H2O of crystn., stable in air, but slowly loses H2O in vacuo at 100.degree., the anhyd. salt being yellow. It deflagrates on a Pt foil. Acidified in concd. aq. soln. at 0.degree. with dil. H2SO4, filtered and washed until neutral, it gives p-nitrophenylnitrosohydroxylamine, p-O2NC6H4N(:O):NOH (IV), m. 75.degree. (decompn.), unstable in light, decompd. by boiling water to HNO2 and p-O2NC6H4NO. Besides III, the following salts of IV were prepd. Phenythydrazine salt, from PhHNNH2 and IV in Et2O, light yellow with pearly luster, m. 96.5.degree. to a red liquid which rapidly decomps., leaving a dark red residue, m. 140.degree.. NH4 salt (p-nitrocupferron), by bubbling dry NH3 through IV in Et2O, egg-yellow, becoming rose or flesh-color at 80.degree., m. 145.degree. to a brown liquid (decompn.), decomps. slowly to ONC6H4NO2. Ag salt, from aq. III and AgNO3, iridescent, stable, but slowly decompd. to Ag and probably PhNO by boiling water. Ba salt, contains 3 mols. of H2O of crystn., cream-yellow, m. 260.degree. to a black liquid (decompn.). Ferric salt, brilliant maroon-red front Me2CO, m. 202.degree. (decompn.); its Me2CO soln. dild. with water gives almost no color with KSCN or with K4Fe(CN)6, while (NH4)2S gives a slowly forming ppt. Cu salt, gelatinous bluegreen from aq. III, gives a green soln. in boiling Me2CO and a dark green soln. in C5H6N, sepg. from C5H6N as very dark green crystals contg. C5H6N; heated to 100.degree. it becomes light blue, darkens at 200.degree. and decomps. at 250.degree.. Ni salt, pptd. on adding aq. NiCl2, to aq. III, as a green-yellow ppt. which redissolves when excess III is still present and reppts. with more NiCl2, gives a very dark green solo. in C6H5N, from which it can be crystd. with a grass-green color, which becomes lighter in air through loss of C6H5N, darkens at 225.degree., in, 249.degree. (decompn.). Co salt, prepd. like the Ni salt, first as a cream-yellow ppt.; from Me2CO it is light reddish yellow with silvery aspect, from C6H5N it is violet-red, darkening at 100.degree. and m. 222.degree. (decompn.). II (1 g.) added slowly to PhNO2: (0.5 g.) suspended in water (20 cc.), heated, concd. at 50-60.degree., more II (1 g.) added, evapd. (with addn. of water to destroy the excess II) to a small vol., filtered and washed with a little water, gives a residue of PhN(:O):NONa, with Na.NO, in the filtrate. Under the same conditions p-MeC6H4Me forms Na nitro-p-tolylhydroxylamine, and dil. alc. m-C6H4(NO2)2 heated 1.5 hrs. at 80.degree. with excess II (3 parts), cooled, evapd. in vacuo

to a small vol., filtered and recrystd. from water gives Na m-nitrophenylnitrosohydroxylamine, m-O2NC6H4N(:O):NONa, yellow, turns brown toward 200.degree., melts at a high temp. to a brown liquid (decompn.). Acidified in water with dil. H2SO4 it yields m-nitrophenylnitrosohydroxylamine, m. 75-6.degree. to a yellowish liquid (decompn.); a large vol. of its, aq. soln. appears green-yellow; it is decompd. by hot dil. H2SO4. to m-ONC6H4 and with HNO2 it forms the m-nitrophenyldiazonium salt. II (5.2 g.) added slowly to p-C6H4(NO2)2 (1.75 g.) in EtOH (75 cc.) and water (30 cc.), heated at 50-60.degree., evapd. in vacuo to 1/2 the vol. and filtered gives a residue of 0.75 g. of p-nitrophenetole, which, purified from EtOH, m. 57-3.degree., forms with HNO2 (d. 1.48) 2,4-dinitrophenetole, m. 86.degree., while from the filtrate is recovered III. II (3.5 g.) added slowly to III (4 g. contg. 5 mols.H2O) in water (50 cc.), heated to 50-60.degree. evapd. in vacuo, filtered, and the residue purified with water and EtOH, gives Na dinitrosophenylenedihydroxylamine (01), reddish yellow, turns brown around 250.degree.; on a Pt foil it blackens and deflagrates. It contains 1 mol. of H2O of crystn. which is liberated in vacuo. Acidified in water at 0.degree. by dil. H2SO4 it forms bis[nitrosohydroxylamine], blackens 85-90.degree., volatilizes when heated in the air and slowly turns yellowish even in vacuo and in darkness. V suspended in water, acidified with dil. H2SO2 and steam-distd. yields nitrous vapors and p-C6H4(NO)2, begins to turn brown at 184.degree., is almost black at 245.degree. but does not melt, is oxidized to p-C6H4(NO2)2 by hot HNO, (d. 1.48). From V is prep. its ferric salt, dark red, turns brown at 200.degree. and decomps. at 215.degree., gives only a slowly forming and very pale blue tint when to its aq. suspension acidified with HCl is added K4Fe(CN)6, unless heated, whereupon the normal blue ppt. appears. With (p-O2NC4H4N:)2 II yields an unidentified dark red nitrosohydroxylamine, the Na salt of which is orange-yellow. Excess (over 2 mols.) II added to hot dil. alc. azoxybenzene, and evapd., yields Ph2N2 and an unidentified orange compd. Hot alc. (p-O2NC6H4)2N2O and II form 20% of (p-O2NC6H4N:)2 and an unidentified nitrosohydroxylamine. II (1.4 g.) added slowly to p-nitroazoxybenzene (0.7 g.) in hot EtOH (70 cc.), let stand 2 hrs., filtered and washed, yields a residue of p-nitroazobenzene, while the filtrate contains an unidentified nitrosohydroxylamine. K (2 g.) dissolved in AmOH (then dild. slightly with water) boiled 48 hrs. with PhN:N(:O)C6H4N(:O):NC6H4N(:O):-NPh (0.4 g,) ppts, 0.3 g, of PhN:NC6H4N:NC6H4N:NPh (VI), while in soln. is found an unidentified violet-red compd. PhN:NC6H4N(:O):NC6H4:NPh and nitroazoxybenzene likewise react with alc. OH to form VI.

CC 10 (Organic Chemistry)

L56 ANSWER 17 OF 18 HCA COPYRIGHT 2003 ACS

17:7888 Original Reference No. 17:1391c-i,1392a-c Selenium nitride. Strecker, W.; Claus, L. Ber., 56B, 362-83 (Unavailable) 1923.

GI For diagram(s), see printed CA Issue.

AB Liquid NH3 reacted vigorously with Se2Cl2 or Se2Br2 with sepn. of red Se. From solns. of the former in CHCl3 or dry C6H6 only traces

of nitride were sometimes observed in the red Se. Solns. in Et_2O at -80.degree. **formed** with **NH3** much red Se and a colorless liquid which became bright yellow during removal of Et_2O and excess NH_3 and deposited bright-brown Se_2NCl and Se_2N_2Br. $SeOCl_2$ in Et_2O soln. in open vessels formed white $SeOCl_2.4NH_3$, decompd. by water to Se and SeN; in a closed tube $SeOCl_2$ and excess of NH_3 were maintained 3 days at room temp. then at 50.degree. for 2 hrs. The tube was cooled in Et_2O-solid CO_2; explosion was caused by opening but most of the product, chiefly SeN, was saved. Solid $SeCl_4$ and $SeBr_4$ react violently with liquid NH_3 with sepn. of Se; in CS_2 suspension $SeCl_4$ becomes deep green, the color vanishing on evapn. of **NH3**; the **product** is a yellow-white amorphous powder, instantly decompd. by water with sepn. of Se and a little SeH_2, which could not be obtained pure. $SeBr_4$ in CS_2 soln. reacts less vigorously than $SeCl_4$ and gives SeN purer and in better yield than Verneuil's method (Bull. soc. chim. [2] 38, 548(1882)). The reaction is not that found by V. for $SeCl_4$ but corresponds to $3SeBr_4 + 16NH_3 = 2SeN + Se + N_2 + 12NH_4Br$. SeN is best prepd. by leading dry cold NH_3 gas into a soln. of 5 g. $SeBr_4$ in 500 cc. absolutely dry CS_2 in a 1-l. Erlenmeyer flask **cooled** by ice-**salt** and protected by a soda-lime tube. The ppt. of SeN, NH_4Cl and Se is filtered, washed with CS_2, placed in a flask, CS_2 removed by dry air, NH_4Cl by water and Se by refluxing with CS_2 and the product dried in air or over H_2SO_4. As solvent for $SeBr_4$ dry C_6H_6 may be used and NH_3 admitted at 5-10.degree.. SeN forms a bright-orange powder, extremely explosive when dry and its instability increases with its degree of purity. It must be kept in cardboard and not in glass as friction of a stopper upon a particle may cause violent detonation. Analysis was by explosion in a special vacuum app. (pictured) and measuring N_2 evolved. Explosion temp. was 160.degree.. A dil. soln. of Br_2 in CS_2 was added to a suspension of SeN in CS_2 and allowed to react 5 days, more Br_2 being added on days 2 and 3; the color become successively yellow, brown, red and the product finally assumed a uniformly dark color; after washing with CS_2 and drying in vacuo it formed a brown-green powder, deliquescent in air, decompd. instantly by water with sepn. of Se, sol. in concd. HNO_3 with decompn., with **NaOH** gives Se and NH_3. Analysis indicated the formula SeN_2Br_4. Br vapor detonates dry SeN but by slow action. In an atm. of CO_2 reaction was begun on several 10-mg. portions for 1 hr. and then continued 4 days in pure Br vapor; the product was first liquid then formed a deep-red powder, which was washed with CS_2 and recrystd. from concd. HBr in deep-red crystals identical with Muthmann's $(NH_4)_2SeBr_6$. Under similar conditions Cl_2 much dild. with CO_2 forms a mixt. which can be exposed to an atm. of Cl_2 and reaches const. wt. in 2 days. The product is bright rose-brown, fairly stable in air, decompd. by water to Se, H_2SeO_3 and HCl. Analysis indicated the formula $SeNCl_3$. Under similar procedure Br_2 with Se_2N_2Br formed $(NH_4)_2SeBr_6$. SeN did not react with solid I or in Et_2O or $CHCl_3$ soln. but exploded on contact with fused I. Mol. wt. detn. was impossible as no solvent could be found. A suspension of NaN_3 in C_6H_6 was boiled with reflux and a soln. of $SeOCl_2$ in C_6H_6 added dropwise. The N_2 evolved was

measured and corresponded to about 0.5 that of the NaN3. The product was 2NaCl.SeO2.H2O. It is concluded from this and other expts. with Se halides and AgN3 that SeN is not comparable to an azide of Se. If the formula Se4N4 is assumed, in analogy with S4N4, the reactions of the nitride are in accord with a structure such as

CC 6 (Inorganic Chemistry)

L56 ANSWER 18 OF 18 HCA COPYRIGHT 2003 ACS

12:8944 Original Reference No. 12:1518i,1519a-i Vulcanization without sulfur according to Ostromuielenskii. Bunschoten, E. (Delft). Chem. Weekblad, 15, 257-68 (Unavailable) 1918.

AB A number of papers have been published by Ostromuislenskii (C. A. 10, 3176-8) on vulcanizing without S; but his results are not stated in quant. terms, and nothing is said about the mechanical properties of the products. Some of his work has been repeated by Stevens (C. A. 11, 1571), who paid more attention to the mechanical properties. Attempts to vulcanize crude rubber with PhNO3 and with m-dinitrobenzene, by heating 30, 60 and 90 min. at 147.degree. on an oil-bath, did not confirm the results described by O. No vulcanization was observed even in the presence of org. **catalysts**, such as "accelerene" (p-ONC6H4NMe2) or "vulcacite" (an **ammonia**-MeCHO **condensation product**), when heated for 30, 60, 90, 120, 150 and 180 min. at 147.degree.. With PbO and PhNO2 some vulcanization took place, but the tensile strength of the product was very low. With PbO and m-dinitrobenzene vulcanization was much more rapid and complete, and the strength was much greater, being highest after 10-15 min. at 147.degree.. When treated longer, up to 2 hrs., the strength steadily decreased. As would be expected, smoked sheet vulcanized more rapidly than crepe in all expts. with only a few exceptions. Conditions were varied to det. the influence of the amt. of PbO or of m-dinitrobenzene, the temp. and the time of treatment. The results are presented in tables. Mixts. of 100 parts of sheet rubber (vulcanization coefficient 4.5) and 8 pts. of PbO were vulcanized at 147.degree. with 1, 2, 3, 4, 6, and 8 pts. of m-dinitrobenzene; and 100 pts. of sheet and 4 pts. of m-dinitrobenzene with 2, 5, 8 and 11 pts. of PbO. Increase of either component increases the velocity of vulcanization. The maximum tensile strength (103 kg. per sq. cm.) was obtained with 8 pts. of PbO and 4 of m-dinitrobenzene. Temps. of 137, 142, 147, 152 and 157.degree. were tried. Rizing temp. greatly increases the velocity of vulcanization, but does not have great influence on the mechanical properties. The time was varied in all the above expts. The best results were obtained with about 15-20 min. treatment. This method might be used instead of the S method if necessary; but the vulcanized rubber deteriorates very rapidly in strength. Rings which were strong when fresh could be easily broken in the hand after 1 month. This does not seem to be due to oxidation, since the Me2CO ext. and the insoly. in C6H6 remain the same even after 6 months. On samples vulcanized only a short time or at a low temp., a white sublimate of m-dinitro. benzene appears after a few days. Attempts were made to prepare ebonite by heating 100 pts. of crude

rubber, 8 pts. of PbO and 10, 15 and 20 pts. of m-dinitrobenzene at 147.degree. for 1, 2 and 3 hrs. No ebonite was formed in any case. The products were leathery, had little strength, and were covered with a layer of yellow crystals which were found to be m-nitroaniline. Since O. regards vulcanization as an oxidation process, mixts. were treated at 147.degree. with 100 pts. of crude rubber and 4 pts. of each of the following: Ae2O5, KSO4, chloride of lime, K2Cr2O7, KClO4, **KNO3**, **NaNO3**, MnO3 and K3Fe(CN)6. In no case was vulcanization complete, but the results were better than with the org. agents in absence of PbO. With a proper **catalyst** these oxidizing agents could probably be made to give good results. To test the theory that vulcanization is due to polymerization, the effect of m-dinitrobenzene on the viscosity of rubber solns. in C6H6 was studied. With 1 g. of rubber and 0.04 g. of m-dinitrobenzene in 100 cc. of C6H6 there was practically 110 effect; but with 0.4 g. of m-dinitrobenzene there was a slight increase of viscosity in the dark and a very rapid decrease when exposed to light. Warming to 65.degree. caused a greater increase in the dark; the decrease in the light was about the same. This indicates depolymerization in light and polymerization in the dark, both accelerated by m-dinitrobenzene. By analogy, vulcanization with S may be regarded as at least partially due to polymerization, accelerated by the S and the heat.

CC 30 (Rubber and Allied Substances)

=> d l57 1-40 ti

L57 ANSWER 1 OF 40 HCA COPYRIGHT 2003 ACS
TI Preparation of 2-pyrrolidone via the condensation of .gamma.-butyrolactone and ammonia in the presence of water using a continuous reactor apparatus

L57 ANSWER 2 OF 40 HCA COPYRIGHT 2003 ACS
TI Process for treating ammonia-containing wastewater

L57 ANSWER 3 OF 40 HCA COPYRIGHT 2003 ACS
TI Preparation of highly-active catalyst for **ammonia synthesis**

L57 ANSWER 4 OF 40 HCA COPYRIGHT 2003 ACS
TI Process for producing amines by reductive amination in the presence of a cobalt catalyst.

L57 ANSWER 5 OF 40 HCA COPYRIGHT 2003 ACS
TI Lignin polymer composition for the treatment of skin problems

L57 ANSWER 6 OF 40 HCA COPYRIGHT 2003 ACS
TI Method for recovery of ammonia from dry solid residues from stack gas scrubbing

L57 ANSWER 7 OF 40 HCA COPYRIGHT 2003 ACS

TI **Ammonia synthesis** catalyst containing cerium oxide

L57 ANSWER 8 OF 40 HCA COPYRIGHT 2003 ACS
TI Destabilizing action of acidic, alkaline, and neutral reagents on emulsions in the form of oil-containing **condensates** from **ammonia manufacture**

L57 ANSWER 9 OF 40 HCA COPYRIGHT 2003 ACS
TI Production of **sodium nitrate**

L57 ANSWER 10 OF 40 HCA COPYRIGHT 2003 ACS
TI Method of obtaining a ferrous catalyst for **synthesis** of **ammonia**

L57 ANSWER 11 OF 40 HCA COPYRIGHT 2003 ACS
TI High-activity spherical catalyst for low-temperature **synthesis** of **ammonia**

L57 ANSWER 12 OF 40 HCA COPYRIGHT 2003 ACS
TI Manufacture of Graham salt with oxidizer

L57 ANSWER 13 OF 40 HCA COPYRIGHT 2003 ACS
TI Polymeric fertilizers

L57 ANSWER 14 OF 40 HCA COPYRIGHT 2003 ACS
TI Sodium nitrite

L57 ANSWER 15 OF 40 HCA COPYRIGHT 2003 ACS
TI Nitrate solutions

L57 ANSWER 16 OF 40 HCA COPYRIGHT 2003 ACS
TI Analysis of boron nitride-boron mixtures produced in a thermal plasma reactor

L57 ANSWER 17 OF 40 HCA COPYRIGHT 2003 ACS
TI Synthesis of indole derivatives. VIII. Synthesis of indole derivatives from corresponding hydrocarbostyril derivatives

L57 ANSWER 18 OF 40 HCA COPYRIGHT 2003 ACS
TI The chemical state of carbon, nitrogen, and oxygen in alkali metals

L57 ANSWER 19 OF 40 HCA COPYRIGHT 2003 ACS
TI Complex fertilizers

L57 ANSWER 20 OF 40 HCA COPYRIGHT 2003 ACS
TI High-concentration nitric oxide

L57 ANSWER 21 OF 40 HCA COPYRIGHT 2003 ACS
TI New barbituric acid **synthesis** in liquid **ammonia** -alkali hydroxide. II. **Condensation** of malonamide derivatives with ethyl carbonate

L57 ANSWER 22 OF 40 HCA COPYRIGHT 2003 ACS
TI Deserpidine, an alkaloid from Rauwolfia

L57 ANSWER 23 OF 40 HCA COPYRIGHT 2003 ACS
TI The reaction between ammonia and transition-metal halides. IV. The reaction of ammonia with vanadium(IV) chloride

L57 ANSWER 24 OF 40 HCA COPYRIGHT 2003 ACS
TI 2,4-Diamino-5-aryl-6-chloropyrimidines

L57 ANSWER 25 OF 40 HCA COPYRIGHT 2003 ACS
TI Production of chlorobromopyridinoamminoplatinum in its three isomeric forms

L57 ANSWER 26 OF 40 HCA COPYRIGHT 2003 ACS
TI The reaction between isatin and ammonia. VI. Oxidation and nitrosation of isamic acid

L57 ANSWER 27 OF 40 HCA COPYRIGHT 2003 ACS
TI Physicochemical investigations in the field of the ammonia-soda process. IV. Kinetics of crystallization of sodium bicarbonate from supersaturated solutions

L57 ANSWER 28 OF 40 HCA COPYRIGHT 2003 ACS
TI Ammonium chloride and alkaline nitrates

L57 ANSWER 29 OF 40 HCA COPYRIGHT 2003 ACS
TI Electric fusion of the **ammonia-synthesis** catalyst

L57 ANSWER 30 OF 40 HCA COPYRIGHT 2003 ACS
TI Some properties of biacetyl monoxime

L57 ANSWER 31 OF 40 HCA COPYRIGHT 2003 ACS
TI The Curtius degradation applied to .alpha.-isoxazolecarboxylic acid and to .alpha.-methyl-.gamma.-isoxazolecarboxylic acid

L57 ANSWER 32 OF 40 HCA COPYRIGHT 2003 ACS
TI Alkali salts

L57 ANSWER 33 OF 40 HCA COPYRIGHT 2003 ACS
TI Separating ammonia from gas mixtures

L57 ANSWER 34 OF 40 HCA COPYRIGHT 2003 ACS
TI The formation of hydrazine during the oxidation of ammonia and during the **synthesis** of **ammonia** in a flame

L57 ANSWER 35 OF 40 HCA COPYRIGHT 2003 ACS
TI The conversion of alkali chlorides into nitrates with simultaneous production of chlorine

L57 ANSWER 36 OF 40 HCA COPYRIGHT 2003 ACS
TI Colorimetric determination of nitrates in water

L57 ANSWER 37 OF 40 HCA COPYRIGHT 2003 ACS
TI Action of cyanogen halides on phenylhydrazine. IV. Transformation into derivatives of o-phenylenediamine

L57 ANSWER 38 OF 40 HCA COPYRIGHT 2003 ACS
TI Alkali inspector's report for 1917

L57 ANSWER 39 OF 40 HCA COPYRIGHT 2003 ACS
TI The ammoniates of silver salts. I, II

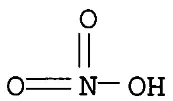
L57 ANSWER 40 OF 40 HCA COPYRIGHT 2003 ACS
TI A Process for Micro-determination of the Constituents of the Blood

=> d 157 3,6,7,8,10,11,19,29,32,33 cbib abs hitstr hitind

L57 ANSWER 3 OF 40 HCA COPYRIGHT 2003 ACS
123:261157 Preparation of highly-active catalyst for **ammonia synthesis**. Sun, Li; Song, Qihong; Wang, Jingtang (Chinese Academy of Sciences, Metal Institute, Peop. Rep. China). Faming Zhuanli Shenqing Gongkai Shuomingshu CN 1099353 A 19950301, 5 pp. (Chinese). CODEN: CNXXEV. APPLICATION: CN 1993-115769 19930827.
AB The title catalyst is manufd. by melting a mixt. of magnetite, alumina, calcium carbonate, and **potassium nitrate** or spent mixed iron oxide catalysts obtained from **ammonia synthesis** and quickly solidifying the melt at a **cooling** speed >100 .degree.K/s.
IT **7664-41-7P**, **Ammonia**, **preparation**
(**prepn**. of highly-active catalyst for **ammonia synthesis**)
RN 7664-41-7 HCA
CN Ammonia (8CI, 9CI) (CA INDEX NAME)

NH_3

IT **7757-79-1**, **Potassium nitrate**, uses
(prepn. of highly-active catalyst for **ammonia synthesis**)
RN 7757-79-1 HCA
CN Nitric acid potassium salt (8CI, 9CI) (CA INDEX NAME)



K

IC ICM C01C001-04
ICS B01J023-74; B01J037-08
CC 49-8 (Industrial Inorganic Chemicals)
Section cross-reference(s): 67
ST magnetite active catalyst **ammonia synthesis**;
alumina active catalyst **ammonia synthesis**;
calcium carbonate catalyst **ammonia synthesis**;
potassium nitrate catalyst **ammonia**
synthesis
IT Catalysts and Catalysis
(prepn. of highly-active catalyst for **ammonia**
synthesis)
IT **7664-41-7P**, **Ammonia**, **preparation**
(**prepn**. of highly-active catalyst for **ammonia**
synthesis)
IT 471-34-1, Calcium carbonate, uses 1309-38-2, Magnetite, uses
1344-28-1, Alumina, uses **7757-79-1**, **Potassium**
nitrate, uses
(prepn. of highly-active catalyst for **ammonia**
synthesis)

L57 ANSWER 6 OF 40 HCA COPYRIGHT 2003 ACS
119:166887 Method for recovery of ammonia from dry solid residues from stack gas scrubbing. Ruegg, Hans (Von Roll AG, Switz.). Eur. Pat. Appl. EP 538647 A1 19930428, 6 pp. DESIGNATED STATES: R: AT, BE, CH, DE, DK, ES, FR, GB, IT, LI, LU, NL, SE. (German). CODEN: EPXXDW. APPLICATION: EP 1992-116513 19920926. PRIORITY: CH 1991-3113 19911024.
AB The waste solids from scrubbing of flue gases are mixed with water, the pH is adjusted to a basic level with an alkali, e.g., CaO, Ca(OH)2, and/or **NaOH**, and the NH3 is steam stripped from the mixt. using the water in the mixt. as to form the steam, preferably in a thin-film evaporator. The **NH3** mixt. is **condensed** and recycled to the process.
IT **7664-41-7P**, **Ammonia**, **preparation**
(**ammonia** recovery from dry solid residues from flue gas
scrubbing for recycling)
RN 7664-41-7 HCA
CN Ammonia (8CI, 9CI) (CA INDEX NAME)

NH_3

IT **1310-73-2P, Sodium hydroxide (**
NaOH), uses
(reagent, ammonia recovery from dry solid residues from flue gas scrubbing for recycling)
RN 1310-73-2 HCA
CN Sodium hydroxide (Na(OH)) (9CI) (CA INDEX NAME)

Na— OH

IC ICM C01C001-02
ICS B01D053-34
CC 59-4 (Air Pollution and Industrial Hygiene)
IT **7664-41-7P, Ammonia, preparation**
(**ammonia** recovery from dry solid residues from flue gas scrubbing for recycling)
IT 1305-62-0P, Calcium hydroxide (Ca(OH)2), uses 1305-78-8P, Calcium oxide (CaO), uses **1310-73-2P, Sodium hydroxide (NaOH)**, uses
(reagent, ammonia recovery from dry solid residues from flue gas scrubbing for recycling)

L57 ANSWER 7 OF 40 HCA COPYRIGHT 2003 ACS
110:102630 **Ammonia synthesis** catalyst containing cerium oxide. Lin, Weiming; Huang, Chuanrong; Gan, Shifan (Huanan Polytechnical College, Peop. Rep. China). Faming Zhuanli Shenqing Gongkai Shuomingshu CN 86107630 A 19880518, 10 pp. (Chinese). CODEN: CNXXEV. APPLICATION: CN 1986-107630 19861105.
AB A catalyst for **NH3 synthesis** is **manufd**. by mixing Fe3O4 93-97, CaO 1.3-2.5, Al2O3 1.3-2.5, K2O 0.3-1.0, and CeO2 0.4-1.0%, m. 1600.degree., adjusting the Fe2+/Fe3+ ratio to 0.55 .+-. 0.02 by adding Fe metal, **cooling** rapidly to room temp., and pulverizing to give particle size 2.2-3.3 mm. Ce(NO3)3, Ce(CO3)2, or Ce(C2O4)2 can replace CeO2; **KNO3** or K2CO3 can replace K2O; and CaCO3 can replace CaO in the manuf. of the catalyst. The catalyst has improved thermal stability, low tendency of poisoning, long working life, and high catalytic activity, even under low temp. and low pressure.
IT **7664-41-7P, Ammonia, preparation**
(**manuf**. of, cerium oxide-iron oxide-contg. catalysts for)
RN 7664-41-7 HCA
CN Ammonia (8CI, 9CI) (CA INDEX NAME)

NH_3

IC ICM B01J023-74

ICS B01J021-04; C01C001-02
CC 67-1 (Catalysis, Reaction Kinetics, and Inorganic Reaction
Mechanisms)
Section cross-reference(s): 49
ST **ammonia synthesis** catalyst iron oxide; cerium
oxide **ammonia synthesis** catalysis
IT Hydrogenation catalysts
(cerium oxide-iron oxide-contg., for **ammonia**
manuf.)
IT 1305-78-8, Calcium oxide, uses and miscellaneous 1306-38-3, Cerium
dioxide, uses and miscellaneous 1317-61-9, Iron oxide (Fe3O4),
uses and miscellaneous 7439-89-6, Iron, uses and miscellaneous
12136-45-7, Potassium oxide, uses and miscellaneous
(catalysts contg., for **ammonia manuf**.)
IT **7664-41-7P**, **Ammonia**, **preparation**
(**manuf**. of, cerium oxide-iron oxide-contg. catalysts
for)

L57 ANSWER 8 OF 40 HCA COPYRIGHT 2003 ACS
110:13042 Destabilizing action of acidic, alkaline, and neutral reagents
on emulsions in the form of oil-containing **condensates**
from **ammonia manufacture**. Andrienko, N. M.;
Drushlyuk, N. S. (USSR). Khimiya i Tekhnologiya Vody, 10(5), 453-7
(Russian) 1988. CODEN: KTVODL. ISSN: 0204-3556.
AB Oil-in-water emulsions in wastewater from **NH3**
manuf. are destabilized by acidic wastewaters. The use of
HCl, **NaOH**, quartz sand, crushed rock, and powd. coke,
slag, lime, coal, and graphite for alk. emulsion breaking is
described.
IT **1310-73-2**, **Sodium hydroxide**, uses and
miscellaneous
(oil-in-water emulsion destabilization by, in **ammonia**
manufg. wastewater treatment)
RN 1310-73-2 HCA
CN Sodium hydroxide (Na(OH)) (9CI) (CA INDEX NAME)

Na—OH

IT **7664-41-7P**, **Ammonia**, **preparation**
(wastewaters from manuf. of, oil-in-water emulsion in,
destabilization of, agents for)
RN 7664-41-7 HCA
CN Ammonia (8CI, 9CI) (CA INDEX NAME)

NH_3

CC 60-2 (Waste Treatment and Disposal)
Section cross-reference(s): 49
ST **ammonia manuf** wastewater emulsion breaking
IT Lime (chemical)

Sand
(oil-in-water emulsion destabilization by, in **ammonia manufg**. wastewater treatment)
IT Slags
Coke
(powd., oil-in-water emulsion destabilization by, in **ammonia manufg**. wastewater treatment)
IT Wastewater
(acidic, oil-in-water emulsion destabilization by, in **ammonia manufg**. wastewater treatment)
IT Wastewater treatment
(emulsion breaking, of oil-in-water emulsions from **ammonia manuf**.)
IT Coal
(powd., oil-in-water emulsion destabilization by, in **ammonia manufg**. wastewater treatment)
IT **1310-73-2**, **Sodium hydroxide**, uses and miscellaneous 7647-01-0, Hydrochloric acid, uses and miscellaneous 7782-42-5, Graphite, uses and miscellaneous
(oil-in-water emulsion destabilization by, in **ammonia manufg**. wastewater treatment)
IT 7631-86-9
(sand, oil-in-water emulsion destabilization by, in **ammonia manufg**. wastewater treatment)
IT 7664-41-7
(wastewater treatment, emulsion breaking, of oil-in-water emulsions from **ammonia manuf**.)
IT **7664-41-7P**, **Ammonia**, **preparation**
(wastewaters from manuf. of, oil-in-water emulsion in, destabilization of, agents for)

L57 ANSWER 10 OF 40 HCA COPYRIGHT 2003 ACS
109:28271 Method of obtaining a ferrous catalyst for **synthesis** of **ammonia**. Dworak, Edward; Golebiowski, Andrzej; Stolecki, Kazimierz (Instytut Nawozow Sztucznych, Pulawy, Pol.). Pol. PL 131490 B1 19851230, 3 pp. (Polish). CODEN: POXXA7. APPLICATION: PL 1980-228423 19801212.
AB In a method for prepn. of these catalysts contg. Fe oxides and promoters, such as Al_2O_3, SiO_2, CaO, K_2O, TiO_2, and others, a melt of Fe with Al and Si is prepd., contg. 50-100% of the metal components of the catalyst. Ferrosilicon can also be used as the raw material for melt prodn. Rods 10-20 mm in diam. are formed from melts and are baked in an O atm. The oxidn. product is **cooled**, crushed into particles <3 mm, and mixed mech. with the remaining oxide-type activators. The suggested proportions of these activators per 92.8 kg of the oxidized melt are: K_2O (as **KNO_3**) 1.4 kg, CaO 1.95 kg, and MgO 0.45 kg. Finally, the total mass is melted again, poured into steel tubs to **cool**, and the solidified alloy is crushed, screened, sepd. into several grain size fractions, and used directly as a ferrous catalyst for **NH_3 synthesis**. In view of the fact that the FeAlSi melt usually contains S, which acts as a catalyst poison, S

is removed from this melt by the addn. of Ce and/or a Ce alloy contg. La, Nd, and Pr, in an amt. of 0.5-2 kg per ton of the melt. Very stable oxysulfides and sulfides of lanthanides formed in this process gather on the surface of the melt and are sepd.

IT **7664-41-7P, Ammonia, preparation**
(**manuf**. of, ferrous catalyst for)

RN 7664-41-7 HCA

CN Ammonia (8CI, 9CI) (CA INDEX NAME)

NH_3

IC B01J023-74; C01C001-04

CC 67-1 (Catalysis, Reaction Kinetics, and Inorganic Reaction Mechanisms)
Section cross-reference(s): 49

IT Hydrogenation catalysts
(ferrous, for **ammonia manuf**.)

IT 1332-37-2, Iron oxide (unspecified), uses and miscellaneous
(catalyst contg., for **ammonia manuf**.)

IT **7664-41-7P, Ammonia, preparation**
(**manuf**. of, ferrous catalyst for)

IT 1305-78-8, Calcium oxide, uses and miscellaneous 1309-48-4, Magnesium oxide, uses and miscellaneous 12136-45-7, Potassium oxide, uses and miscellaneous 13463-67-7, Titanium dioxide, uses and miscellaneous
(promoter, for ferrous catalyst for **ammonia manuf**.)

L57 ANSWER 11 OF 40 HCA COPYRIGHT 2003 ACS

108:115165 High-activity spherical catalyst for low-temperature **synthesis** of **ammonia**. Wang, Wenxiang; Feng, Dinghao; Guo, Yiqun; Li, Limin; Li, Guanpu (Zhengzhou University, Peop. Rep. China). Faming Zhuanli Shenqing Gongkai Shuomingshu CN 85101605 A 19860917, 5 pp. (Chinese). CODEN: CNXXEV. APPLICATION: CN 1985-101605 19850401.

AB The title catalyst comprises Al2O3 1.9-2.6, K2O 0.9-1.3, CaO 0.7-1.3, SiO2 <0.45, S <0.01, P <0.04%, and balance Fe3O4, and is prepd. by melting a mixt. of magnetite 100, Al2O3 2-1, **KNO3** 2-8, CaCO3 1.7, and metallic Fe 7 parts in an elec. furnace, dispersion, **cooling**, and heat treatment.

IT **7664-41-7P, Ammonia, preparation**
(**manuf**. of, iron oxide-based catalyst for)

RN 7664-41-7 HCA

CN Ammonia (8CI, 9CI) (CA INDEX NAME)

NH_3

IC ICM B01J023-78
ICS B01J035-08; B01J037-08

CC 49-8 (Industrial Inorganic Chemicals)

Section cross-reference(s): 67
ST iron oxide catalyst **ammonia synthesis**; alumina catalyst **ammonia synthesis**; calcium oxide catalyst **ammonia synthesis**; potassium oxide catalyst **ammonia synthesis**
IT Hydrogenation catalysts
(iron oxide-based, for **ammonia manuf**.)
IT 1317-61-9, Iron oxide (Fe_3O_4), uses and miscellaneous
(catalysts contg., for **ammonia manuf**.)
IT 1305-78-8, Calcium oxide, uses and miscellaneous 12136-45-7, uses and miscellaneous
(catalysts from iron oxide contg., for **ammonia manuf**.)
IT **7664-41-7P, Ammonia, preparation**
(**manuf**. of, iron oxide-based catalyst for)

L57 ANSWER 19 OF 40 HCA COPYRIGHT 2003 ACS
61:57576 Original Reference No. 61:10002a-b Complex fertilizers. Geiersberger, Karl; Nees, Hugo; Vorster, Fritz (Chemische Fabrik Kalk G.m.b.H.). US 3132935 19640512, 12 pp. (Unavailable). PRIORITY: DE 19570314.
AB Mixed fertilizers contg. $CaHPO_4$, $NH_4H_2PO_4$, NH_4NO_3, and **KNO_3** are prepd. in 4 steps: (1) treat phosphate rock with 50-60% HNO_3 (6.4-7.4 moles HNO_3/mole P_2O_5) **cool**, and sep. the cryst. $Ca(NO_3)_2.4H_2O$ to give a CaO/P_2O_5 mole ratio <2.3 in the liquor; (2) react the $Ca(NO_3)_2.4H_2O$ with NH_3 and CO_2 and sep. NH_4NO_3 from the pptd. $CaCO_3$; (3) either of 2 processes: (a) add 30-70% by wt. of the sepd. NH_4NO_3 soln. from step 2 to the mother liquor of step 1, neutralize with NH_3, and add K salt equiv. to 1-3.5 moles K_2O/mole P_2O_5 or (b) distill 50-75% of the HNO_3 from the mother liquor of step 1, conc. the resulting dil. HNO_3 (using the result again in step 1), add all of the NH_4NO_3 from step 2, neutralize with NH_3, and add K salt as in 3a; and (4) mix 1 part of step 3 product with 1-3.5 parts of recycled dry final product, granulate, and dry.
NCL 071039000
CC 73 (Fertilizers, Soils, and Plant Nutrition)
IT Fertilizers
(manuf. of, by phosphate rock acidulation by HNO_3 and reaction of **produced** $CaNO_3$ with **NH_3** and CO_2)

L57 ANSWER 29 OF 40 HCA COPYRIGHT 2003 ACS
32:51708 Original Reference No. 32:7219i,7220e-i Electric fusion of the **ammonia-synthesis** catalyst. Telegin, V. G.; Sidorov, N. V. J. Applied Chem. (U. S. S. R.), 11, 588-96(in French 596) (Unavailable) 1938.
AB The Larson and Richardson method (cf. C. A. 19, 3003, and U. S. 1,554,008, C. A. 19, 3571) was used. Natural magnetite (Fe 70.6, FeO 27.3, Al_2O_3 1.4 and SiO_2 0.9%) and pure Al_2O_3 (4.5%) and **KNO_3** (5.3%) were used as initial materials. The reduction and test for activity of the catalyst was carried out in the previously described app. under the following conditions: (a) the reduction: 5 ml. of the catalyst (1-2 mm. mesh) was reduced at atm.

pressure with N2-H2 mixt. passing with the vol. velocity of 20,000 l./l. of catalyst for 24 hrs. at 500.degree.; (b) the activity was detd. by the NH3 content in the gas passing from the reaction chamber at 200 and 300 atm., with the vol. velocity of 15,000 at 400.degree., 450.degree., 475.degree., 500.degree. and 525.degree.. Three catalysts prepd. by electro-fusion for 20, 40 and 60 min. had approx. the same activity, because the 1st stage of the homogenation of the catalyst, i. e., soln. of the promoting oxides in the mass of the Fe oxides, proceeded with a considerable velocity. However, the formation of more complex combination between Fe oxides and promoters guaranteeing its activity at high temp. and prolonging its work depended on the time of existence of the alloy in the liquid state, as was shown by the increase of activity of the catalyst at 525.degree. and 300 atm. The duration of **cooling** of similar catalysts for 16, 2 hrs. and 6 min., resp., had practically no effect on its activity, although tempering of the catalyst somewhat decreases its activity. The conditions of crushing had no effect on the activity of the catalyst. The catalyst obtained consisted of 2 layers: (I) inner, homogeneous mass of fused magnetite and the promoters with inclusions of the gas bubbles, and (II) outer, where the transition from completely fused layer to caked material was observed. The analysis for the degree of Fe oxidation in both layers disclosed that II was oxidized more than I. Addn. of **KNO3** oxidized the catalyst, while introducing the K2O into the alloy. Seventeen references.

CC 18 (Acids, Alkalies, Salts, and Other Heavy Chemicals)

L57 ANSWER 32 OF 40 HCA

25:25267 Original Reference No. 25:2818c-d Alkali salts. (Chemieverfahren Ges). FR 697069 19300603 (Unavailable). APPLICATION: FR .

AB **KNO3** and Na2CO3 are prepd. A lye, obtained by mixing CaSO4 in a mother liquor contg. Na2CO3 and sepg. the CaCO3 thus formed, is treated with a crude sylvinitic salt of potash and **NH3** which **produces** glaserite and KCl. The K2SO4, obtained by treating this mixt. with water, is treated with CaCO3, previously obtained, and HNO3 which gives CaSO4 and **KNO3**. The lye sepd. from the glaserite and KCl is **cooled** to remove a part of the NH4Cl and is then submitted to the Solvay process.

CC 18 (Acids, Alkalies, Salts, and Sundries)

L57 ANSWER 33 OF 40 HCA

25:25266 Original Reference No. 25:2818a-c Separating ammonia from gas mixtures. Frowein, Friedrich; Rahlfs, Erich (I. G. Farbenind. A.-G.). DE 519225 19290111 (Unavailable). APPLICATION: DE .

AB The mixts. are treated with mother liquors which contain metal salts capable of combining with NH3 and which cannot easily be worked up in any other way. A suitable mother liquor is obtained in the manuf. of **KNO3** from Ca(NO3)2 and KCl. The liquor comprises CaCl2 with some **KNO3**, and when treated while **cooling** with gases contg. NH3, e. g., with **NH3**

synthesis products, CaCl2.6NH3 is pptd. After
sepg. the ppt., the residual soln. may be returned to the first
stage of the **KNO3** manuf., or worked up separately to
recover **KNO3**. The pptd. CaCl2.6NH3 is decompd. by heat.
Cf. C. A. 25, 2250.

CC 18 (Acids, Alkalies, Salts, and Sundries)

=> d l58 1-27 ti

L58 ANSWER 1 OF 27 HCA COPYRIGHT 2003 ACS
TI Red iron oxide nanoparticles and manufacture process thereof

L58 ANSWER 2 OF 27 HCA COPYRIGHT 2003 ACS
TI Production of a sulfonated phenolic resin with reduced free formaldehyde and impregnation of paper therewith

L58 ANSWER 3 OF 27 HCA COPYRIGHT 2003 ACS
TI Recovery and purification of rhodium

L58 ANSWER 4 OF 27 HCA COPYRIGHT 2003 ACS
TI Novel heterocyclic compounds

L58 ANSWER 5 OF 27 HCA COPYRIGHT 2003 ACS
TI Study of some Claisen reactions occurring with ethyl diethoxyacetate

L58 ANSWER 6 OF 27 HCA COPYRIGHT 2003 ACS
TI .alpha.-Nitrilo-.alpha.'-carboxydiisopropyl ether and its derivatives

L58 ANSWER 7 OF 27 HCA COPYRIGHT 2003 ACS
TI Verdazyls, a new class of cyclic N-containing radicals

L58 ANSWER 8 OF 27 HCA COPYRIGHT 2003 ACS
TI Solarium alkaloids. XXIII. Synthesis of solarium alkaloids from 16.beta.-hydroxypregnane derivatives

L58 ANSWER 9 OF 27 HCA COPYRIGHT 2003 ACS
TI Optical activity and magnetic rotation as contributors to the chemical bonding and electronic configuration of the period I elements

L58 ANSWER 10 OF 27 HCA COPYRIGHT 2003 ACS
TI O,N-Substituted hydroxylamines. I. O-(.beta.-Hydroxyethyl)hydroxylamine, a new type analog of .beta.-aminoethanol, and derivatives. Investigations of synthetic medicinals

L58 ANSWER 11 OF 27 HCA COPYRIGHT 2003 ACS
TI Alkaloids of Senecio mikanioides Otto. Sarracine and sarracine N-oxide

L58 ANSWER 12 OF 27 HCA COPYRIGHT 2003 ACS
TI Synthesis of 1-ethyl-1,2,3,5-tetrahydro-10-methyl-7,8-methylenedioxy-5-oxobenzo[f]pyrrocoline, a degradation product of lycorine

L58 ANSWER 13 OF 27 HCA COPYRIGHT 2003 ACS
TI Condensation of dialkali metal .beta.-diketones with ketones or aldehydes to form hydroxy .beta.-diketones. Dehydration products. Equilibrium factors

L58 ANSWER 14 OF 27 HCA COPYRIGHT 2003 ACS
TI N-6-Puryl derivatives of biogenic amines and amino acids

L58 ANSWER 15 OF 27 HCA COPYRIGHT 2003 ACS
TI Some alkaloids of Iboga and of Voacanga (Apocynaces)

L58 ANSWER 16 OF 27 HCA COPYRIGHT 2003 ACS
TI The structure of artificial rosins. VII. Oxidative degradation of the methylene-nitrogen bridges in phenol-hexamethylenetetramine condensates

L58 ANSWER 17 OF 27 HCA COPYRIGHT 2003 ACS
TI Codeine and codiene intermediates

L58 ANSWER 18 OF 27 HCA COPYRIGHT 2003 ACS
TI Benzhydrylalkylamines; contribution to the analysis of the mitosis-inhibiting action of colchicine

L58 ANSWER 19 OF 27 HCA COPYRIGHT 2003 ACS
TI Terramycin. X. The structure of Terramycin

L58 ANSWER 20 OF 27 HCA COPYRIGHT 2003 ACS
TI The enol methyl ether of benzoylacetone

L58 ANSWER 21 OF 27 HCA COPYRIGHT 2003 ACS
TI Cyclic polyolefins. XI. Carbonyl-bridged compounds derived from the adduct of 2-carbethoxycyclohexanone and acrolein

L58 ANSWER 22 OF 27 HCA COPYRIGHT 2003 ACS
TI Reaction between acetone and **ammonia**: the **formation** of pyrimidine compounds analogous to the aldoxanes of Spath

L58 ANSWER 23 OF 27 HCA COPYRIGHT 2003 ACS
TI Sulfanilylurea derivatives

L58 ANSWER 24 OF 27 HCA COPYRIGHT 2003 ACS
TI .gamma.-Triazines. XVIII. Synthesis of m-tolyl-, p-tolyl- and benzyldiaminotriazine

L58 ANSWER 25 OF 27 HCA COPYRIGHT 2003 ACS
TI **Manufacture** of synthetic **ammonia** at Oppau,

Germany. I, II

L58 ANSWER 26 OF 27 HCA COPYRIGHT 2003 ACS
TI **Manufacture** of synthetic **ammonia** at Oppau,
Germany. I, II

L58 ANSWER 27 OF 27 HCA COPYRIGHT 2003 ACS
TI Synthesis of .gamma.-piperidone

=> d l58 25,26 cbib abs hitstr hitind

L58 ANSWER 25 OF 27 HCA COPYRIGHT 2003 ACS
15:6669 Original Reference No. 15:1191c-i,1192a-c **Manufacture** of synthetic **ammonia** at Oppau, Germany. I, II. Anon. Chem. Met. Eng., 24, 305-8,347-50 (Unavailable) 1921.

AB After Haber completed Le Chatelier's work by detg. the conditions for max. combination of N and H, it required 10 years' work to solve the problems of making large amts. of pure N and H, extg. the NH3 from the mixt., conversion of the ammonia into readily marketable products and the devizing of suitable app. A mixt. is made of water gas and producer gas. Steam is added and the whole is passed over a **catalyzer** at 400-500.degree.. CO and H2O form CO2 and H. The CO2 is dissolved in water at 25 kg. and traces of CO2 and CO are removed by passing the gas through **NaOH** and ammoniacal copper formate solns. N from liquefied air brings the nitrogen vol. to one-third that of H. This mixt. is then heated to about 500.degree., compressed at 200 kg. and subjected to the action of a second **catalyzer**. The **NH3 produced** is absorbed by water. The residual gases with fresh additions are passed again through the **catalyzer**. The NH3 may be oxidized to HNO3, converted into NH4NO3, NH4Cl, (NH4)2SO4, etc. Mixed ammonia and **potash** salts are made up into special fertilizers. The gas producers and water-gas generators are described with operating data. The producer and water gases pass separately through turbine-type washers and through cyclones to complete their purification. They are then metered and mixed in the ratio of two parts of water gas to one of producer gas. **Catalytic** oxidation of Co. The gas mixt. receives 76 g. of steam per cu. m. as exhaust from the turbo-ventilators, and is satd. with moisture by passing through jets of water at 95.degree. on its way to the **catalyzing** chambers. The **catalyst**, a mixt. of FeO and Cr2O3, is kept at 500.degree.. The gas mixture after treatment contains about 30% CO2 and 1.5% CO. The gases are compressed in five stages. When at 27 kg. they are treated in absorption towers with water under pressure. The CO2 is reduced to less than 1%. CO is removed by treating the gases with ammoniacal cuprous formate soln. under 200 kg. pressure in towers of forged steel tubes. The remaining CO2 is next removed by treatment with **NaOH** soln. at the same pressure. About 8 cu. m. is consumed hourly. II. **Catalysis** of the N and H Mixture. Increase in pressure aids the union of N and H, whereas increase in temp.

lowers the yield of NH3 but increases the rate of reaction. The theoretical maximum yield of NH3 at 600.degree. under a pressure of 200 atm. is from 8 to 9%. The gases are circulated continuously and the NH3 is absorbed by water as fast as formed. H diffuses through steel at temps. above 450.degree., combines with carbon, forming methane, and leaves cavities in the metal. The **catalyzer** is designed so that H diffusing from the inner tube meets N in an outer tube. The inner tube is 12 m. high, 1.10 m. external diam., and 12 cm. thick in wall. It and the outer tube are of steel. The gases on leaving the **catalyzers** are **cooled** to ordinary temp. and treated with water at 200 atm. pressure. At the start the **catalyzers** are heated to 500.degree. by means of a mixture of air and hydrogen injected at 200 atm. and ignited by a hot Pt wire. The heat of reaction then maintains the required temp. A 25% soln. of NH4OH is produced, and the gas is removed by treatment with steam and stored in a gas holder over water covered with oil. Ammonia Oxidation. The **catalyzer** used is thought to be a mixt. of oxides of iron, manganese and chromium agglomerated with BiCl3. It is prepd. in granules 5 to 8 mm. in diam. and forms layers about 5 cm. deep on perforated shelves. NH3 and air are mixed and passed over the contact mass in the ratio of 36.8 cu. m. of the former to 46.6 of the latter. Gas furnaces supply heat to the oxidation furnaces. The oxidized gases supply heat to eight waste-heat boilers. Each of sixteen furnaces oxidizes about four tons of NH3 daily with 80% efficiency. Absorption of Nitrous Vapors. If absorbed by water in towers, the vapors yield 50% nitric acid. If they are passed into Na2CO3 soln. a mixt. of nitrate and nitrite is obtained.

CC 18 (Acids, Alkalis, Salts, and Sundries)

L58 ANSWER 26 OF 27 HCA

15:6668 Original Reference No. 15:1191c-i,1192a-c **Manufacture** of synthetic **ammonia** at Oppau, Germany. I, II. Anon. Technique moderne, 12, 449-60 (Unavailable) 1920.

AB After Haber completed Le Chatelier's work by detg. the conditions for max. combination of N and H, it required 10 years' work to solve the problems of making large amts. of pure N and H, extg. the NH3 from the mixt., conversion of the ammonia into readily marketable products and the devizing of suitable app. A mixt. is made of water gas and producer gas. Steam is added and the whole is passed over a **catalyzer** at 400-500.degree.. CO and H2O form CO2 and H. The CO2 is dissolved in water at 25 kg. and traces of CO2 and CO are removed by passing the gas through **NaOH** and ammoniacal copper formate solns. N from liquefied air brings the nitrogen vol. to one-third that of H. This mixt. is then heated to about 500.degree., compressed at 200 kg. and subjected to the action of a second **catalyzer**. The **NH3 produced** is absorbed by water. The residual gases with fresh additions are passed again through the **catalyzer**. The NH3 may be oxidized to HNO3, converted into NH4NO3, NH4Cl, (NH4)2SO4, etc. Mixed ammonia and **potash** salts are made up into special fertilizers. The gas producers and water-gas generators are

described with operating data. The producer and water gases pass separately through turbine-type washers and through cyclones to complete their purification. They are then metered and mixed in the ratio of two parts of water gas to one of producer gas. **Catalytic** oxidation of Co. The gas mixt. receives 76 g. of steam per cu. m. as exhaust from the turbo-ventilators, and is satd. with moisture by passing through jets of water at 95.degree. on its way to the **catalyzing** chambers. The **catalyst**, a mixt. of FeO and Cr2O3, is kept at 500.degree.. The gas mixture after treatment contains about 30% CO2 and 1.5% CO. The gases are compressed in five stages. When at 27 kg. they are treated in absorption towers with water under pressure. The CO2 is reduced to less than 1%. CO is removed by treating the gases with ammoniacal cuprous formate soln. under 200 kg. pressure in towers of forged steel tubes. The remaining CO2 is next removed by treatment with **NaOH** soln. at the same pressure. About 8 cu. m. is consumed hourly. II. **Catalysis** of the N and H Mixture. Increase in pressure aids the union of N and H, whereas increase in temp. lowers the yield of NH3 but increases the rate of reaction. The theoretical maximum yield of NH3 at 600.degree. under a pressure of 200 atm. is from 8 to 9%. The gases are circulated continuously and the NH3 is absorbed by water as fast as formed. H diffuses through steel at temps. above 450.degree., combines with carbon, forming methane, and leaves cavities in the metal. The **catalyzer** is designed so that H diffusing from the inner tube meets N in an outer tube. The inner tube is 12 m. high, 1.10 m. external diam., and 12 cm. thick in wall. It and the outer tube are of steel. The gases on leaving the **catalyzers** are **cooled** to ordinary temp. and treated with water at 200 atm. pressure. At the start the **catalyzers** are heated to 500.degree. by means of a mixture of air and hydrogen injected at 200 atm. and ignited by a hot Pt wire. The heat of reaction then maintains the required temp. A 25% soln. of NH4OH is produced, and the gas is removed by treatment with steam and stored in a gas holder over water covered with oil. Ammonia Oxidation. The **catalyzer** used is thought to be a mixt. of oxides of iron, manganese and chromium agglomerated with BiCl3. It is prepd. in granules 5 to 8 mm. in diam. and forms layers about 5 cm. deep on perforated shelves. NH3 and air are mixed and passed over the contact mass in the ratio of 36.8 cu. m. of the former to 46.6 of the latter. Gas furnaces supply heat to the oxidation furnaces. The oxidized gases supply heat to eight waste-heat boilers. Each of sixteen furnaces oxidizes about four tons of NH3 daily with 80% efficiency. Absorption of Nitrous Vapors. If absorbed by water in towers, the vapors yield 50% nitric acid. If they are passed into Na2CO3 soln. a mixt. of nitrate and nitrite is obtained.

CC 18 (Acids, Alkalis, Salts, and Sundries)

=> d his 159-

FILE 'HCA' ENTERED AT 12:15:21 ON 12 JUN 2003

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L59          232 S (ALK# OR ALKALIN#)(A)NITRATE#
L60            5 S L7 AND L59
L61            4 S L60 NOT (L56 OR L57 OR L58)
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=> d l61 1-4 ti

L61 ANSWER 1 OF 4 HCA
TI Guanidine-based solid propellants and gas generators for use in airbags and fire extinguishers

L61 ANSWER 2 OF 4 HCA
TI Electrochemical processing of **alkaline nitrate** and nitrite solutions

L61 ANSWER 3 OF 4 HCA
TI Metabolism of soil fungi

L61 ANSWER 4 OF 4 HCA
TI Action of Ammonium and **Alkaline Nitrates** and Nitrites as well as the Corresponding Salts of some Substituted Ammonium Compounds toward Reducing Agents